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NSWC CRANE
5090.3a

DEPARTMENT OF THE NAVY
CRANE DIVISION
NAVAL SURFACE WARFARE CENTER
300 HIGHWAY 361
CRANE INDIANA 47522-5001

IN REPLY REFER TO:

5090
Ser 095/2256

01 AUG 2002

2-1-2003

U.S. Environmental Protection Agency, Region V
Waste, Pesticides, & Toxics Division
Waste Management Branch
Illinois, Indiana, and Michigan Section
ATTN: Mr. Peter Ramanauskas (DW-8J)
77 West Jackson Blvd.
Chicago, IL 60604

Dear Mr. Ramanauskas:

Crane Division, Naval Surface Warfare Center (NSWC Crane) submits the Final RCRA Facility Investigation (RFI) Quality Assurance Project Plan (QAPP) for Solid Waste Management Units 12/14, 13/14, 16/16, and 19/00 (Mine Fill A, Mine Fill B, Cast High Explosive-Building 146 Incinerator Complex, and Pyrotechnic Test Areas, respectively). Two copies are provided as enclosure (1). The permit required Certification Statement is provided as enclosure (2).

NSWC Crane point of contact is Mr. Thomas J. Brent, Code 09510, telephone 812-854-6160.

Sincerely,

James M. Hunsicker

JAMES M. HUNSICKER
Director, Environmental
Protection Department
By direction of the Commander

Encl:
(1) Final RFI QAPP for SWMUs 12/14, 13/14, 16/16, and 19/00
(2) Certification Statement

Copy to:
ADMINISTRATIVE RECORD
SOUTHNAVFACENGCOM (Code ES32) (w/o encl)
IDEM (Doug Griffin)
TTNUS (Ralph Basinski) (w/o encl)

TETRA TECH NUS, INC.
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PITT-07-2-062

July 26, 2002

Project No. 3994

Mr. William Gates (Code 1864)
Commander, Southern Division
Naval Facilities Engineering Command
2155 Eagle Drive, P.O. Box 190010
North Charleston, SC 29419-9010

Reference: CLEAN Contract No. N62467-94-D-0888
Contract Task Order No. 0166

Subject: Final Quality Assurance Project Plan (QAPP) for
Mine Fill A (SWMU 12), Mine Fill B (SWMU 13),
Cast High Explosives Fill/B-146 Incinerator (SWMU 16), and
Pyrotechnic Test Area (SWMU 19) RCRA Facility Investigation (RFI)

Dear Mr. Gates:

The subject final QAPP is enclosed. This QAPP has been revised in response to U.S. EPA Region 5 comments. Attachment 1 contains the responses to U.S. EPA Region 5 comments. The responses have been revised in accordance with Navy comments received via e-mail and telephone conversations.

Please contact Ralph Basinski at (412) 921-8308 (e-mail basinskir@ttnus.com) regarding any questions or comments.

Sincerely,

Ralph R. Basinski

Ralph R. Basinski
Task Order Manager

Enclosures

cc: Mr. Tom Brent, NSWC Crane (letter, attachment, 5 copies of QAPP)
Ms. Debbie Wroblewski, TtNUS, Inc. (letter only)
Mr. Mark Perry, TtNUS, Inc. (1 copy of letter, attachment, and unbound QAPP)
Mr. Ralph Basinski, TtNUS, Inc. (1 copy of letter, attachment, and QAPP)
Crane Library (1 copy of QAPP)
File Copy – CTO 0166 (letter and attachment)

ATTACHMENT I

**RESPONSE TO US EPA COMMENTS ON
THE QUALITY ASSURANCE PROJECT PLAN (QAPP) FOR
THE RCRA RFI INVESTIGATION AT
SWMUs 12, 13, 16, & 19 - REVISION 0 - DATED FEBRUARY 2002
NAVAL SURFACE WARFARE CENTER CRANE
CRANE, INDIANA**

***"Comment 1:* Where was the NPDES outfall mentioned in the first full sentence at the top of page 1-2 located and why is it no longer monitored or needed?"**

Response: The NPDES outfall mentioned in the 4th paragraph of Section 1.0 was not actually at the OTA. This outfall was actually an in-stream monitoring point established in 1984 below the OTA (i.e., above Lake Gallimore) as a checkpoint for monitoring runoff from the majority of NSWC Crane. As this outfall also monitored non-point sources, this type of monitoring point was contrary to the scope of the NPDES program because the NPDES program only regulated point-source discharges. Because this outfall was not actually at the OTA, references to this outfall have been removed from the Quality Assurance Project Plan for Mine Fill A (SWMU 12), Mine Fill B (SWMU 13), Cast High Explosives Fill/B146 Incinerator (SWMU 16), and Pyrotechnic Test Area (SWMU 19) RCRA FRI Investigation (QAPP).

The 3rd sentence in the 4th paragraph of Section 1.01 has been revised as follows to remove all reference of this outfall from the QAPP:

"No analytical data are available for the Pyrotechnic Test Area."

***"Comment 2:* The first bullet point in Section 1.1.1. refers to spring water. Is this a reference to spring water screening levels or actual springs present at the SWMUs? If the latter, where are these springs located?"**

Response: The reference is to spring water screening levels and not to actual springs at these sources.

No changes have been made to the QAPP in response to this comment.

***"Comment 3:* Should the first sentence of the 2nd Paragraph in Section 1.1.3. read pre-pre-QAPP?"**

Response: The Navy agrees. The 1st sentence of the 2nd paragraph of Section 1.1.3 has been revised as follows:

"Additional guidance regarding development of this QAPP was obtained through a pre-pre-QAPP meeting held on July 11, 2001 via conference call."

***"Comment 4:* The second full paragraph on page 1-15 refers to an "existing closed pit" receiving wash-out water at Mine Fill A. Is this being checked for integrity? Also, the following paragraph references a the No. 2 fuel oil tank. Have there been any releases from this tank?"**

Response: This pit is no longer "existing." This pit was removed when the building and equipment at the B-3110 treatment plant was installed in 1984 and 1985. The No. 2 fuel oil tank no longer exists, and there is no record of any leaks from this tank.

For clarification, the 13th sentence in the 12th paragraph of Section 1.3.2 has been revised as follows:

"Overflow from the baffled tank then flowed via floor drains to a pit outside of the building, which has since been removed."

Additionally, for further clarification, the 13th paragraph of Section 1.3.2 has been revised as follows:

"Heat used to generate the steam for the melting and steam-out operations was generated by boilers that burned No. 2 fuel oil. These boilers were located in Building 150 outside MFA. The No. 2 fuel oil tank no longer exists. There are no records of any leaks from the No. 2 fuel oil tank."

***"Comment 5:* What is the integrity of the sumps collecting scrubber wastewater mentioned on page 1-16?"**

Response: The scrubber wastewater collection sumps are contained within Building B-3111. Sump integrity is checked whenever steam-out operations occur. The sumps have not shown signs of leakage.

For clarification, the following text has been added after the 12th sentence of the 16th paragraph of Section 1.3.2:

"The sumps are checked for integrity whenever steam-out operations occur. The sumps have not shown signs of leakage."

***"Comment 6:* The 3rd paragraph on page 1-17 seems to be incomplete. The 4th paragraph refers to the asphalt pad at the southern end of MFA. Is sediment/surface water sample 12SW/SD09 located at the drainage ditch which originates at the outfall of the pump system at the pad? If so, it should be analyzed for the same constituents as soil boring 12SB01."**

Response: First, the 19th paragraph of Section 1.3.2 was a typographical error that has been removed from the QAPP.

Next, the unidentified asphalt pad (UAP) pump system outfall could not be found during the site visit. However, there is a narrow topographic depression that slopes along a straight line from the UAP towards the swale to the west of the UAP. To address the potential for contaminant migration from the UAP, sample 12SW/SD09 was located in the area from the UAP toward the swale.

The Navy agrees that, with the exception of VOCs, sample 12SW/SD09 should be analyzed for the same constituents as soil boring 12SB01. Therefore, explosives and semivolatiles analyses have been added to the existing analytical suite for this sample. Because the potential for VOCs to exist in surface water and shallow sediment is low, due to volatilization, VOCs analyses are not recommended for this sample.

For clarification, the 4th bullet in THE 4th paragraph in Section 1.4.1 Subsection SWMU 12 has been revised as follows:

- *"SVOCs (surface and subsurface soils, surface water, and sediments) - SVOCs, including polycyclic aromatic hydrocarbons (PAHs), may have been released into soils and accumulated near the UAP. The potential exists for migration toward downstream surface water conveyances."*

Finally, Tables 3-11 through 3-14 have been modified to reflect the semivolatile organic compound (SVOC) analysis for sample 12SW/SD09.

***"Comment 7:* Has the Navy discovered any futher information on the surface ponds and/or settling basins mentioned by Halliburton NUS at MFA & MFB?"**

Response: The Navy and Crane Army Ammunition Activity (CAAA) has no additional information on these surface ponds or settling basins for the MFA (SWMU 12—Mine Fill A) and MFB (SWMU 13—Mine Fill B).

No changes have been made to the QAPP in response to this comment.

ATTACHMENT I

"Comment 8: **Page 1-15 Section 1.3.3. mentions that some of the pollution control equipment added to MFA was not added to MFB. What areas/buildings/processes do not have this equipment and are there any consequences? What are spill potentials (current or historical) at areas like Building 173 (mine degreasing) mentioned on pg 1-18? Were any inspections done on these areas to look for evidence of releases?"**

Response: The MFA has pollution control equipment on B-160 that included the "pinkwater" treatment plant (B-3110), the wet scrubbers (B-3111), and an electrostatic precipitator (ESP) (B-155) to control tar emissions during bomb loading. Because there is no current bomb loading nor are there any future plans to do bomb loading at MFB, a "pinkwater" treatment plant, wet scrubbers, and ESP are not necessary.

For clarification, the following text has been added to the end of the 1st paragraph of Section 1.3.3:

"Some pollution control equipment to control air and water emissions installed at the MFA was not added at the MFB. This equipment included pinkwater treatment, wet scrubbers, and an electrostatic precipitator (ESP). This equipment controls emissions from bomb loading. The MFB does not have the capabilities for bomb loading and there are no plans to use MFB for bomb loading. Therefore, the lack of this equipment is not viewed to result in chemical release to the environment."

Building B-173 is no longer active (e.g., no treatment activities occur at Building B-173). Therefore, currently, there is no spill potential at Building B-173.

For clarification, the 12th sentence in the 5th paragraph of Section 1.3.3 has been revised as follows:

"Building 173 formerly used to degrease mines is no longer used. There are no records of historical spills in this area."

CAAA has no records of historical spills in this area.

No changes have been made to the QAPP in response to this portion of this comment.

"Comment 9: **Referring to Page 1-19, top half of page, to what levels were PCBs remediated?"**

Response: During the remediation process for explosives-contaminated soils at MFB, soils containing polychlorinated biphenyls (PCBs) in addition to explosives were also removed. No specific PCB remediation goals were established. Confirmation sampling indicated that the remaining soils contained PCBs in excess of 10 milligrams per kilogram (mg/kg).

Additionally, the reference in the last sentence of the 7th paragraph in Section 1.3.3 to Building 177 should have been to Building 171.

The last sentence of the 7th paragraph in Section 1.3.3 has been replaced as follows:

"During the course of the remediation of explosive-contaminated soils at the MFB, soils containing PCBs were removed. No specific remediation goal for PCB was established for this soil removal. Confirmation sampling indicated that the remaining soils contained PCBs in excess of 10 mg/kg. Some residual PCB contamination still remains near Buildings 166 and 171 (Halliburton NUS, 1992a)."

"Comment 10: **Why isn't VOC included in the MFB constituent list bullets on page 1-38 due to the degreasing operations? Note that naphtha is mentioned at the top of page 1-38. Although it is stated in Table 1-3, p. 1 of 2 that VOCs will be analyzed in samples for SWMU 13, discussion of VOCs sampling isn't found on pp. 1-38 to 1-39. This is perhaps a minor oversight."**

Response: Similarly to MFA, there were no known pathways of release from the building where the operations took place. Therefore, volatile organic compounds (VOCs) were not included in the MFB constituent list.

No changes have been made to the QAPP in response to this comment.

***"Comment 11:* The last sentence of the 2nd paragraph on page 1-21 mentions TCE at high concentrations in settling basins - are these the same as the sumps that were addressed under the Interim Measure for SWMU 16?"**

Response: Yes, these settling basins are the same as the sumps that were addressed under the Interim Measures for SWMU 16.

No changes have been made to the QAPP in response to this comment.

***"Comment 12:* There is a reference to chromium VI on page 1-21 (as well as in other places), which suggests that use of an analytical test for hexavalent chromium would be appropriate for certain specific objectives (i.e. as opposed to only using methods for reporting total chromium, such as methods based on SW-846 - 6020)."**

Response: Analytical methods for chromium will only involve testing for total chromium. The reference to hexavalent chromium could not located in the QAPP. A search of the QAPP was conducted to determine whether the referenced page number (1-21) could be incorrect. An electronic search of the entire QAPP text and Table 1-7 could not locate the existence of a reference to hexavalent chromium.

No changes were made to the QAPP to address this comment.

***"Comment 13:* Referring to page 1-36, please note that if metals will be reported in sediments the AVS-SEM data would be of most significance to ecologists (as indicated on page 1-37). Does Crane indeed propose to report both total metals as well as AVS-SEM metals separately on SWMU 12 sediments samples?"**

Response: Yes, Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS-SEM) metals will be reported separately from total metals. For clarification, the following text has been added at the end of Section 1.4.1 Subsection SWMU 12, 3rd paragraph, 8th bullet; Subsection SWMU 13, 3rd paragraph, 10th bullet; Subsection SWMU 16, 3rd paragraph, 12th bullet; and Subsection SWMU 19, 3rd paragraph, 12th bullet:

> *"AVS/SEM metals will be reported separately from total metals."*

***"Comment 14:* Referring to the first bullet on page 1-38, it is mentioned that metals are not of concern based on IM sampling. How do the IM residential cleanup levels compare to the RBTLs noted in Table 1-7 and background soil metals levels? Would levels of metals detected during the IM trigger inclusion as a COPC under the decision diagram of Figure 1-13? If so, will the Navy consider these values when performing risk assessment? This would also be true for MFA."**

Response: First it should be noted that some of the risk-based target levels (RBTLs) listed on Table 1-7 are ecological values and; therefore, cannot be directly compared to the interim measures (IM) residential cleanup levels which are based on human health. However, the ecological values are generally lower than the IM cleanup levels. A comparison of the human health proposed RBTLs for residential soil presented in Appendix B of the QAPP with the IM residential cleanup levels indicates that the values are approximately the same. Any differences are due to the RBTLs being obtained from the most recent United States Environmental Protection Agency (U.S. EPA) Region 9 PRG table whereas the IM cleanup levels were taken from an older US EPA Region 9 PRG table.

It is correct that the levels of metals detected during the IM would trigger the metal's inclusion as a chemical of potential concern (COPC) in the decision diagram and the values for that metal would be considered in the risk assessment. An examination of the post-excavation data indicates that only arsenic would exceed its RBTL. However, this initial examination indicates that the concentrations of arsenic appear to be within background levels. This conclusion will be tested by more rigorous statistical testing that will be performed in the risk assessment. A description of the background comparison methodology is address in Appendix C, Section C.1.2.1 of the QAPP.

No changes have been made to the QAPP in response to this comment.

"Comment 15: **Referring to the SVOC bullet on page 1-38 what is meant by "integrators of contamination"?"**

Response: Sediments often have finer grain size than soils and represent surface soils that have been transported to and mixed in the surface drainages. The fine grain size enhances adsorption of contaminates. Hence, sediments represent contamination that comes from a wider area than an individual soils sample location.

For clarification, the last sentence of the 3rd bullet in the 3rd paragraph of Section 1.4.1 Subsection SWMU 13 has been revised as follows:

- *"SVOCs (surface and subsurface soils, sediments, surface water, and ground water) – SVOCs, including PAHs, may have been released directly to surface or subsurface soils near the therminol boilers. Sediments will be used as integrators of contamination to determine whether further investigation is necessary because they represent the accumulation of surface water runoff from a relatively large area."*

"Comment 16: **Referring to the PCB bullet on page 1-40, it is stated that PCBs "may have been released directly to surface or subsurface soils with subsequent migration to sediments." Will sediments at SWMU 16 be analyzed for PCBs? If so, this should be noted in the parenthesis for the PCB bullet."**

Response: Sediment will not be analyzed for PCBs. Should PCBs be found in soil samples at levels that could indicate potential sediment PCB contamination, a case-by-case determination will be made as to whether or not it is necessary to collect additional sediment samples for PCBs analysis in selected areas.

No changes were made to the QAPP in response to this comment.

"Comment 17: **Please clarify the phrase, "..and potentially surface water and ground water.)" found in the 3rd bullet on page 1-42. When will this be known, definitively?"**

Response: Surface water was inadvertently included in the VOCs analytical scheme. Therefore, the 3rd bullet of the 2nd paragraph of Section 1.4.1 Subsection SWMU 19 has been revised as follows:

- *"VOCs (surface and subsurface soils and potentially ground water) – These compounds may have been released directly to surface or subsurface soils near the cook-off testing areas with potential migration to other media."*

"Comment 18: **The term 'laboratory threshold value - 'TV's', appearing in Table 1-7 should be defined. Furthermore the definition of 'TV' and how it relates to other detection limits should be explained in Sections 1.4, and 1.4.1.2."**

Response: The laboratory commonly does not report analyte concentrations that are less than the laboratory reporting limits (RLs) on practical quantitation limits (PQLs). Because the risk-based target levels are so low, the Navy requested that the laboratory report to concentrations that are less than the RLs. At first, the request was for the laboratory to report to the method detection limits (MDLs), as currently described in this QAPP. Upon reflection the laboratory decided that the MDLs were not necessarily realistic for the matrices to be analyzed for this project. However, the laboratory agreed that the concentrations less than reporting limits could generally be detected. Therefore, the laboratory agreed to compromise by reporting analyte concentrations as low as threshold values that fall between the MDLs and the RLs. The threshold values are intended to replace the laboratory MDLs, but the description of their origin was inadvertently omitted from the text. Therefore, the 2nd paragraph of Section 1.4 has been rewritten into two paragraphs as follows:

"All field parameter results greater than or equal to method detection limits (MDLs) will be reported. Field parameters not detected will be reported at the MDL. MDLs for field parameters are based on method or test kit capabilities and specifications. All laboratory results greater than or equal to threshold values (TVs) will be reported. Target parameters not detected will be reported at the TV. TVs for laboratory parameters are based on method capabilities and specifications. They reflect the computed method detection limit that has been adjusted to reflect what the laboratory realistically can expect to achieve on a routine basis for the environmental matrices to be analyzed. This accounts for day-to-day variations in MDLs that the laboratory has experienced in the past, plus other factors such as the recognition that MDLs were computed from data generated in matrices other than those at SWMUs 12, 13, 16, and 19.

Analytical results for analytes that are less than applicable MDLs (or TV) will be reported with a 'U' flag. The 'U' flag signifies that the parameter was analyzed for but was not detected at a concentration greater than or equal to the MDL (or TV). Analytical results that are between the MDL (or TV) and the reporting limit (RL) will be reported with a 'J' flag. Laboratory TVs are based in part on best professional judgment and on statistical computations in accordance with 40 Code of Federal Regulations (CFR) Part 136, Appendix B. The CFR requires the MDL to be computed as the standard deviation of replicate analyses, with results multiplied by the appropriate Student's t-value. Refer to Laucks SOP LTL-1011 for a mathematical computation of instrument detection limit (IDL)/ MDL. Sample-specific laboratory TVs will be computed for each sample to account for variations in the TV that are caused by factors such as sample moisture content, the size of the sample aliquot used in the analysis, and dilutions. The actual TV used will be an estimate of the MDL achievable in actual sample matrices. This estimate will be based on the laboratory's computed MDLs and the laboratory's best estimate of the effect of actual sample matrices on these computed values. This approach will frequently result in a TV that is greater than the computed MDL but less than the practical quantitation limit (PQL). It is viewed to be a reasonable compromise between using MDLs that are unrealistically low and PQLs that reflect the quality of the concentration estimate rather than the ability to detect the presence of an analyte in a sample."

***"Comment 19:* In the first paragraph on page 1-48, please clarify for which COPCs background comparisons will be made to investigational data (metals only)?"**

Response: The 23 U.S. EPA Target Analyte Metals plus lithium, strontium, thorium, and tin were analyzed under NSWC Crane Basewide Background Soil Investigation (TtNUS, 2001a). The metals to be analyzed under this CTO 166 investigation are a subset of those 27 metals; therefore, all metals analyzed under this CTO 166 investigation will undergo background comparisons as described in the 2nd paragraph of Section 1.4.4.2 of this QAPP. The "background investigation report," provides additional detail on the identification of various NSWC Crane soil groups, statistical treatment of the metals concentration data and how data comparisons may be made to the background data. It should be noted that COPCs are not technically compared to background concentrations because COPCs are selected only after the background comparisons are made. Organic contaminants are assumed not to occur naturally for the purpose of COPC selection so the concentrations in background media are assumed to equal zero. The 2nd paragraph of Section 1.4.4.2 establishes, prior to the start of the investigation, the background data that will be used as the background comparisons for metals.

For clarification, the 4th sentence of the 2nd paragraph of Section 1.4.4.2 has been revised as follows:

"For metals, the background concentrations will be represented by soil data from the NSWC Crane Basewide Background Soil Investigation (TtNUS, 2001a)."

"Comment 20: **In Table 1-3, p. 1 of 2, what does the term, "select spls (i.e. samples)" mean? What might be the rationale for the second footnote of this table where it is stated that "ground water samples will be analyzed for dissolved metals if NTUs less than 10 cannot be achieved during sampling"?"**

Response: The term "select spls" means select samples. "Select spls" indicates that not all the samples that are collected for an environmental medium will be analyzed for every parameter. For clarification, a footnote has been added to Table 1-3 that defines "spls." For further terminology clarification, an acronym list has been added to the QAPP.

The 2nd footnote in Table 1-3 refers to ground water samples that are filtered to remove suspended solids prior to sample collection. All ground water samples are analyzed for total metals. However, turbidity [as measured by nephelometric turbidity units (NTUs)] is an indicator of the amount of suspended solids that are present in ground water. A high turbidity indicates that the ground water collected from a well has not yet reached equilibrium with the formation water. If a sample is highly turbid (e.g., above a 10 NTU), a significant portion of the metals present may be attributable to the solids in the ground water. In these cases, a filtered sample is collected to determine whether the metals are present in the dissolved or solid form.

No change has been made to the QAPP in response to this comment.

"Comment 21: **Should use of an U.S. EPA method for measuring hexavalent chromium in site media of concern be added to Table 1-7? The note "TBD" should be updated in the case of Laucks' HPLC method for picric acid and picramic acid, as indicated in Table 1-7. What is the meaning of a "TV" in Table 1-7, and which will be reported as a detection limit (i.e. a "TV" or a "RL")?"**

Response: Regarding hexavalent chromium, please refer to the Comment 12 response. In Table 1-7, the to be determined (TBD) items for picric and picramic acid have been revised for the Laboratory TV to 0.5 micrograms per liter (µg/L) for the aqueous matrix and to 17 µg/kg for the solid matrix. Additionally, in Table 1-7, the TBD items for picric and picramic acid have been revised for the Laboratory RL to 1 µg/L for the aqueous matrix and to 33 µg/kg for the solid matrix.

Please refer to the response to Comment 18 for a discussion of the threshold value (TV) and the meaning of "TV."

"Comment 22: **Expand on what is meant by "soil replacement areas that are part of the bioremediation facility" mentioned in the second paragraph of page 3-6. Are these the compost backfilled areas? If so, are these sampled for nitrates?"**

Response: Backfill composted material was used for the "soil replacement areas that are part of the bioremediation facility." The composted backfill material was analyzed for cyclotrimethylenetrinitramine (RDX), cyclotetramethylenetetranitramine (HMX), and trinitrotoluene (TNT) prior to being returned to the site. No analysis was conducted for nitrates. A further discussion of nitrate analyses is contained in the response to Comment 27.

No changes have been made to the QAPP in response to this comment.

"Comment 23: **Referring to the settling basins noted in the third paragraph on page 3-6, have there**

been any overflows which might require surface samples here?"

Response: There were several releases of explosives contaminated water to surface soil in the late 1980s and early 1990s. Therefore, there is a potential for energetics and nitrates to be present at the settling basins. To determine impacts, if any, from settling basin overflows, the following changes have been made to the QAPP:

1. Tables 3-2 and 3-3 have been modified to include eight additional surface soil samples (12SS04, 12SS05, 12SS06, 12SS07, 12SS08, 12SS09, 12SS10, and 12SS11) for SWMU 12.

2. The last paragraph of Section 3.4.1.1 has been revised as follows:

"Four soil borings each will be installed along the edges of settling basins located at structures 3110 (12SB04 through 12SB07) and 3037 (12SB08 through 12SB11) to evaluate whether leakage may have occurred. Each boring will be sampled at one surface and two subsurface depth intervals above the water table; specific subsurface depths will be determined in the field based on encountering waste or obvious contamination during drilling. If no waste or obvious contamination is encountered during drilling, then the shallow subsurface sample will be collected from a depth that is representative of the middle depth of the settling basin. The deep subsurface sample will be collected from a depth that is representative of the bottom of the settling basin, but not below the water table."

3. The 3rd sentence of the 2nd bullet of the 4th paragraph of Section 1.4.1 Subsection SWMU 12 has been revised as follows:

"Soils near the UAP and settling basins will be sampled and tested for explosives."

***"Comment 24:* For Cast High soil samples (Section 3.4.1.3.), expand on why subsurface depth is "to be determined"."**

Response: Because the depth at which contamination occurs varies for each sample location, a preset sampling depth cannot be established. Therefore, during drilling, it is necessary to make a field determination at each sample location based upon encountering wastes or obvious contamination where the subsurface soil sample is to be collected (e.g., the "to be determined" subsurface soil sampling depth).

For clarification, the 1st paragraph of Section 3.4.1.3 has been revised as follows:

"As previously discussed in Section 1, soil contamination exists in the incineration ash disposal areas south of Building 146, and shell casings are present on the ground surface in the parking areas surrounding Building 146. Some of the ash has been removed; however, contamination may still be present. Therefore, 39 additional soil borings (16SB01 through 16SB39) are proposed to be installed within and immediately outside these areas to determine whether any residual contamination is present. One surface soil sample (0 to 2 feet bgs) and one subsurface soil sample (depth to be determined based on encountering waste or obvious contamination during drilling) will be collected from each boring. If no waste or obvious contamination is encountered during drilling, then the subsurface soil sample will be collected immediately above the water table."

For additional clarification, a sentence has been added to the end of 2nd paragraph of Section 3.4.1.3 as follows:

"The shallow soil sample will be collected from a depth that is representative of the middle depth of each sump and the deep sample will be collected from a depth that is representative of the bottom of each sump, but not below the water table."

"Comment 25: **Referring to the second paragraph of section 3.4.1.3., the sediment and soil samples are very close to one another as shown on Figure 3-3 (e.g., 16SB40 and 16SW/SD07). EPA suggests adding another sediment sample further down the ditches after the sump discharge because of long term historical discharges to those ditches."**

Response: To determine long-term historical discharges to these ditches after the sump discharge, the following changes have been made to the QAPP:

1. Figure 3-3 has been modified with the addition of four sediment samples (16SD08, 16SD09, 16SD10, and 16SD11).

2. Tables 3-13 and 3-14 have been modified to include four additional sediment samples.

3. A 3rd sentence has been added to the 1st paragraph of 3.4.3.3 as follows:

"A total of seven surface water / sediment samples (16SW/SD01 through 16SW/SD07) are proposed for this SWMU in the ditches that exit this SWMU. Two of the sample locations (16SW/SD02 and 16SW/SD07) are located at the discharge point of the sumps located at the SWMU. A total of four sediment samples (16SD08 through 16SD11) will be collected to determine the impact of long-term historical discharges, if any. This SWMU is also located at a typographic high and the contamination results will be evaluated in respect to background in the same manner as Mine Fills A and B. All surface water/sediment at the SWMU is derived from within the SWMU boundary, and the collection of SWMU-specific background samples and associated evaluation is not appropriate."

"Comment 26: **Referring to section 3.4.1.4., why are there no subsurface borings near saltwater wave tank at the rocket range? Why is boring 19SB01 not located near fast-cook off tank as is 19SB02 (underground brine tank)?"**

Response: The exclusion of a soil boring at the saltwater wave tank located at the rocket range was an oversight. An additional boring (19SB07) will be added to the scope of work. The boring will be sampled for the same depth intervals and analytical suites as boring 19SB02. The following changes have been made to the QAPP:

4. Figure 3-4 has been modified with the addition of boring 19SB07 at the Rocket Range.

5. Tables 3-2 and 3-3 have been modified to include sampling boring 19SB07. Additionally, Table 3-2 has been revised to indicate that the depth for the subsurface soil sample in boring 19SB02 is to be determined.

6. The 4th paragraph of 3.4.1.4 has been revised as follows:

"Both surface and subsurface soil samples will be collected during Round 1 from soil borings at seven locations (19SB01 through 19SB07). One of the soil borings (19SB01) will be located in the vicinity of the fast cook-off tank where burnable fuel is pumped underground from a remote tank to the cook-off tank, then ignited. Soil borings 19SB02 and 19SB07 will be located adjacent to an underground brine tank (not identified on Figure 3-4) and saltwater wave tank, respectively, that are used to test underwater signal flares. The subsurface soil samples from these borings will be collected at a depth representing the tank bottoms, but not below the water table. Soil borings 19SB03 through 19SB06 will be located in a sand trap area that was the landing area for the rocket range."

Additionally, Boring 19SB01 that had been located near the fast cook off tank sampling perimeter, has been re-located to near the cook off tank as detailed on Figure 3-4.

"Comment 27: **For MFA/MFB groundwater sampling (Table 3-4), there is no mention of nitrates sampling. It may be good to be consistent between what is identified in tables 1-3 to 1-5 and tables 3-2 though 3-5 and 3-11 through 3-14."**

Response: Table 3-4 contains a summary of ground water samples to be collected for laboratory analysis. Nitrate analysis for ground water is addressed in Table 3-1 (Field Screening and Measurements). Nitrates will be analyzed in the field.

No changes have been made to the QAPP in response to this comment.

"Comment 28: **In sections 3.4.3.1 & 3.4.3.2. referring to MFA/MFB sediment sampling, EPA suggests taking samples further off the SWMU (i.e., downstream sediment) to assess any impacts from the long term historical discharges to these drainage ditches."**

Response: To determine long-term historical discharges to these drainage ditches from SWMUs 12 and 13, the following changes have been made to the QAPP:

For SWMU 12:

1. Figure 3-1 has been modified with the addition of eight sediment samples (12SD16, 12SD17, 12SD18, 12SD19, 12SD20, 12SD21, 12SD22, and 12SD23) to determine the impacts, if any, of long-term historical discharges associated with SWMU 12 to these drainage ditches.

2. Tables 3-13 and 3-14 have been modified to include eight additional sediment samples.

3. A 2nd sentence has been added to the 1st paragraph of 3.4.3.1 as follows:

   "A total of 15 surface water / sediment samples (12SW/SD01 through 12SW/SD15) are proposed for this SWMU. A total of eight sediment samples (12SD16 through 12SD23) will be collected to determine the impacts, if any, of long-term historical discharges. This SWMU is located at a topographic high point and all surface water that exists at the SWMU originated within the SWMU boundary. The surface water/sediment samples are located in all apparent surface water conveyances at the SWMU along the SWMU boundary to address the majority of surface water runoff. All of these samples are intended to address SWMU related contamination. Therefore, it will be concluded that organic contamination, if present in this medium would be site related. Organic contamination results would be subject to comparison to regional background data sets to determine site related contamination."

For SWMU 13:

1. Figure 3-2 has been modified with the addition of nine sediment samples (13SD14, 13SD15, 13SD16, 13SD17, 13SD18, 13SD19, 13SD20, 13SD21, and 13SD22) to determine impacts, if any, of the long-term historical discharges associated with SWMU 13 to these drainage ditches.

2. Tables 3-13 and 3-14 have been modified to include nine additional sediment samples.

3. A 3rd sentence has been added to the end of the 1st paragraph of 3.4.3.2 as follows:

   "The sampling approach at Mine Fill B is similar to the approach taken at Mine Fill A. A total of 13 surface water/sediment samples (13SW/SD01 through 13SW/SD13) are proposed for this SWMU. Background will be evaluated in the same manner as the Mine Fill A. A total of nine sediment samples (13SD14 through 13SD22) will be collected to determine the impacts, if any, of long-term historical discharges."

***"Comment 29:* For SWMU #19, EPA suggests adding 2 SW/SD samples to creeks running into Boggs Creek from the "Alternate Site" noted on Figure 3-4."**

Response: To determine impacts, if any, of long-term historical discharges to these drainage ditches, the following changes have been made to the QAPP:

1. Figure 3-4 has been modified with the addition of two surface water / sediment samples (19SW/SD26 and 19SW/SD27).

2. Tables 3-11, 3-12, 3-16, and 3-14 have been modified to include two additional surface water / sediment samples.

3. The 1st paragraph of 3.4.3.4 has been revised as follows:

   "As previously discussed, the Pyrotechnic Test Area lies within and along both sides of the floodplain of Boggs Creek. A total of 29 surface water/sediment samples (19SW/SD01 through 16SW/SD27) are proposed for this SWMU, which includes sampling in ditches which are tributaries to Boggs Creek located in proximity to treatment areas. Upstream samples are also proposed, including samples 19SW/SD01 and 19SW/SD06 for background comparison."

***"Comment 30:* For SWMU #19, has the highest flood elevation been determined and are the flood plain soils samples placed appropriately?"**

Response: Information is not available for the highest flood elevation at SWMU 19 including the Rocket Range. However, according to information obtained from site personnel, all of the lowland areas are subject to flooding. The number and placement of flood plain soil samples was based on this information.

No changes have been made to the QAPP in response to this comment.

***"Comment 31:* The Navy should consider using the Encore samplers for analysis of VOCs in sediments per method 5035."**

Response: Regarding Table 3-15, soil and sediment samples collected for volatile organic compound (VOCs) analysis will be collected and containerized in EnCores™ samplers. Footnote 2 has been added to Table 3-15 as follows:

"Sediment samples collected from areas with no standing water will be collected and containerized in this manner. Sediment samples collected from areas under standing water will be containerized using a using a 4 ounce clear wide mouth glass jar and cooled to 4°C."

Additionally, a 7th sentence has been added to the 4th paragraph of Section 3.4.3 and to the Table 3-15 footnotes as follows:

"For volatile fractions only, sediment samples collected from areas with no standing water will be collected and containerized in EnCore™ samplers as noted in Table 3-15."

ATTACHMENT I

Miscellaneous changes to the QAPP not directly associated with response to U.S. EPA comments are as follows:

I. Technical changes:

Analytical laboratory standard operating procedures (SOPs) that have been revised since the draft QAPP was issued have been included as follows:

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)

LAUCKS TESTING LABORATORIES

SOP NUMBER	REV #	QAPP	TITLE
LTL-1020	2	126(1)	Integration of IC, GC, HPLC and GCMS Peaks
LTL-2001	6	126(1)	Waste Segregation and Disposal
LTL-3011	2	126(1)	Extraction Method for Chlorinated Herbicides in Soil (8151A by 3550B)
LTL-3161	5	166(2)	Extraction Method for Ordnance Compounds in Soils (8330)
LTL-8019	4	166(2)	Determination of Non-Halogenated Volatile Organics by SW 8015B
LTL-8084	5	166(2)	Analysis of Organochlorine Pesticides and PCBs by SW 846 Methods SW 8081A and 8082
LTL-8151	4	126(1)	Analysis of Chlorinated Herbicides by 8151A
LTI-8265	4	166(2)	Determination of Volatile Organic Compounds by Method 8260B
LTL-8277	2	166(2)	Determination of Polynuclear Aromatic Hydrocarbon Compounds by Selective Ion Monitoring (SIM) Method 8270C
LTL-8279	3	166(2)	Determination of Semivolatile Organic Compounds by Method 8270C and 8270D
LTL-8303	5	166(2)	Determination of Picric and Picramic Acid
LTL-8330	11	126(2)	Determination of Nitroaromatics and Nitramines by SW-846 Method 8330
LTL-9104	3	166(2)	Preparation and Analysis of Total and Amenable Cyanide According to EPA Methods 335.1, 335.3, and SW 846 9012A
LTL-9110	8	166(2)	Determination of Anions by Ion Chromatography by Method 300.0A or 9056
LTL-9113	2	166(2)	pH in Soil and Waste Samples Using SW846 Method 9045C
LTL-9116	6	166(2)	The Determination of Total Organic Carbon in Soil/Sediment Samples Using Modified SW-846 Method 9060

TRIANGLE LABORATORY

SOP NUMBER	REV #	QAPP	TITLE
2.04	6	126(2)	Nonconformance
5.01	10	126(2)	Sample Receipt, Identification, Handling, and Storage
DSP 105	16	126(2)	Extraction of PCDD/PCDF from Solids (not tissue) – 8290
DSP 161	19	166(1)	Extraction of PCDD/PCDF from Water for Methods 1613, 8290, and 551
DHR 182	9	126(2)	PCDDs and PCDFs by HRGC/HRMS – Method 8290
DPA 104	12	126(2)	Data Package Assembly and Shipping

ATTACHMENT I

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)

NSWC CRANE LABORATORY

SOP NUMBER	REV #	QAPP	TITLE
CR4052-PD-0100	3	166[1]	Document Control
CR4052-PD-0150	4	166[1]	Record Retention
CR4052-PD-0200	3	166[1]	Preventive Maintenance
CR4052-PD-0600	2	166[1]	Customer Reports

1 "Quality Assurance Project Plan for Mine Fill A (SWMU 12), Mine Fill B (SWMU 13), Cast High Explosives Fill/B146 Incinerator (SWMU 16), and Pyrotechnic Test Area (SWMU 19) RCRA FRI Investigation," NSWC Crane, February 2002.

2 "Quality Assurance Project Plan for Ammunition Burning Grounds Little Sulphur Creek and Jeep Trail Resource Conservation and Recovery Act Phase III RCRA Facility Investigation," NSWC Crane, April 2001.

II. Non-technical changes:

A. An Acronym List has been added to the QAPP.
B. Various punctuation and capitalization errors have been corrected throughout the QAPP.
C. Various acronym definitions have been added to the first usage of a term in the QAPP.
D. Various redundant acronym definitions have been eliminated from the QAPP.

Quality Assurance Project Plan

for

Mine Fill A (SWMU 12)
Mine Fill B (SWMU 13)
Cast High Explosives Fill/
B146 Incinerator (SWMU 16) and
Pyrotechnic Test Area (SWMU 19)
RCRA Facility Investigation

Volume 1 - Text and Appendices A - H

Naval Surface Warfare Center
Crane

Crane, Indiana

Southern Division
Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Order 0166

February 2003

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QUALITY ASSURANCE PROJECT PLAN
FOR
MINE FILL A (SWMU 12)
MINE FILL B (SWMU 13)
CAST HIGH EXPLOSIVES FILL/
B146 INCINERATOR (SWMU 16) AND
PYROTECHNIC TEST AREA (SWMU 19)
RCRA FACILITY INVESTIGATION

NAVAL SURFACE WARFARE CENTER, CRANE
CRANE, INDIANA

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ACRONYMS

%	percentage
%R	percent recovery
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
ALPEC	Ammunition Loading and Production Engineering Center
amsl	above mean sea level
ANOVA	ANaylsis Of VAriance
ASCO	Ammunition Supply Control Office
AVCO	thermal coating called AVCO
AVS/SEM	Acid Volatile Sulfide/Simultaneously Extracted Metals
AWQC	federal ambient water quality criteria
BERA	baseline ecological risk assessment
bgs	below ground surface
BRA	baseline risk assessment
CAAA	Crane Army Ammunition Activity
CEC	cation exchange capacity
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CLP	contract laboratory program
CMS	corrective measure study
CN	alpha-chloroacetophenone
COC	chain-of-custody
COPCs	chemicals of potential concern
CTO	Contract Task Order
DPT	direct-push technology
DQOs	data quality objectives
EDQLs	U.S. EPA Region 5 ecological data quality levels
Ei	ranks of the site samples
EMR	environmental monitoring reports
ESP	electrostatic precipitator
EPCs	exposure point concentrations
ERA	ecological risk assessment
ESM	environmental site manager

EU	exposure unit
FOL	field operations leader
FTMR	field task modification request
GC	gas chromatograph
GC/MS	gas chromatograph/mass spectrometer
H0	null hypothesis
HA	alternate hypothesis
HASP	Health and Safety Plan
HBX	high blast explosion
HI	hazard index
HMX	cyclotetramethylenetetranitramine
HPLC	high-performance liquid chromatograph
HSM	health and safety manager
IAS	initial assessment survey
ICP	inductively coupled plasma
ICP/MS	inductively coupled plasma/mass spectroscopy
IDEM	Indiana Department of Environmental Management
IDL	instrument detection limit
IDLs	instrument detection limits
IDW	investigation-derived waste
IMR	interim measures reports
LCS	laboratory control sample
MCLC	mine clearing line charges
MCLs	federal maximum contaminant levels
MDL	method detection limit
MDLs	method detection limits
MEK	methyl ethyl ketone
MFA	SWMU 12 - Mine Fill A
MFB	SWMU 13 - Mine Fill B
mg/kg	milligrams per kilogram
mg/L	milligrams per liter
MK	Morrison-Knudson Corporation
mL/minute	milliliters per minute
MNA	monitored natural attenuation
mph	miles per hour
MS/MSD	matrix spike and matrix spike duplicate

MSDs	matrix spike duplicates
MSs	matrix spikes
NAD	Naval Ammunition Depot
NAD83	1983 North American Datum
NFA	no further action
NFESC	Naval Facilities Engineering Service Center
NFESC	Naval Facilities Engineering Services Command
NIST	National Institute of Standards and Technologies
NOAA	National Oceanic and Atmospheric Administration
NPDES	National Pollutant Discharge Elimination System
NSWC	Naval Surface Warfare Center
NSWC	Naval Surface Warfare Center
NTUs	nephelometric turbidity units
NWSC	Naval Weapons Support Center
ORP	oxidation reduction potential
OSHA	Occupational Safety and Health Administration
OTA	ordnance test area
PAHs	polycyclic aromatic hydrocarbons
PARCC	precision, accuracy, representativeness, comparability, and completeness
PCB	polychlorinated biphenyls
PDSs	post-digestion spikes
PE	performance evaluation
pg/g	picograms per gram
pg/L	picograms per liter
PID	photo-ionization detector
PETN	pentaerythritoltetranitrate
PM	project manager
PPE	personal protective equipment
PQL	practical quantitation limit
PRGs	U.S. EPA Region 9 preliminary remediation goals
PTA	pyrotechnic test area
PVC	polyvinyl chloride
QA	quality assurance
QA/QC	quality assurance/quality control
QAM	quality assurance manager
QAO	quality assurance officer

QAPP	quality assurance project plan
RBSLs	risk-based screening levels
RBTLs	risk-based target levels
RCRA	Resource Conservation and Recovery Act
RDX	cyclotrimethylenetrinitramine
RFI	facility investigation
RLs	reporting limits
RPD	relative percent difference
RPM	remedial project manager
RQAC	quality assurance coordinator
SAP	sampling and analysis plan
SDG	sample delivery group
select spls	select samples
SERA	screening-level ecological risk assessment
SOUTHDIV	Naval Facilities Engineering Command
SOPs	standard operating procedures
SOWs	statements of work
SSLs	soil screening levels
SSO	site safety officer
SVOCs	semivolatile organic compounds
SWMU 12/14	Mine Fill A
SWMU 13/14	Mine Fill B
SWMU 16/16	Cast High Explosives Fill
SWMU 19/00	Pyrotechnic Test Areas
SWMUs	solid waste management units
TCE	trichloroetheylene
TCLP	toxicity characteristic leaching procedures
tetrytol	TNT and Tetryl
TNT	trinitrotoluene
TOC	total organic carbon
TolTest	TolTest Incorporated
TOMs	task order managers
TtNUS	Tetra Tech NUS, Inc.
TVs	threshold values
U.S. EPA	United States Environmental Protection Agency
UAP	unidentified asphalt pad

USACE	United States Army Corp of Engineers
USDA	United States Department of Agriculture
USGS	United States Geological Service
UXO	unexploded ordnance
VOCs	volatile organic compounds
WRS	Wilcoxon Rank-Sum
WWII	World War II

1.0 PROJECT DESCRIPTION

This Quality Assurance Project Plan (QAPP) is being prepared as part of the Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) Phase III work plan. The RFI is being conducted in accordance with applicable RCRA corrective action requirements, which include the need to conduct RFIs at the Naval Surface Warfare Center (NSWC) Crane in Crane, Indiana. Investigation requirements are specified in the NSWC Crane RCRA hazardous waste management permit, originally issued by the United States Environmental Protection Agency (U.S. EPA) on December 29, 1989 and recently renewed on October 18, 2001 (IDEM, 2001). The RFI will provide data on select organic and inorganic chemical concentrations in surface soils, subsurface soils, sediment, surface water, and ground water at four solid waste management units (SWMUs). The four SWMUs addressed in this QAPP are SWMU 12 - Mine Fill A (MFA), SWMU 13 - Mine Fill B (MFB), SWMU 16 - Cast High Explosives Fill/B146 Incinerator, and SWMU 19 - Pyrotechnic Test Area.

Mine Fills A and B are nearly identical units that were used for the production of large mines, depth charges, rocket heads, aerial bombs, and projectiles. In the past, explosives powders discharged from roof vents and accumulation on the roofs were washed down to the ground, resulting in the contamination of soils. Wastewaters containing explosives were previously discharged into ditches. An interim removal action was conducted in which explosive-contaminated soils were removed for bioremediation of explosives. Treated soils were then placed onto the areas from which the contaminated soils were removed.

The Cast High Explosives Fill/B-146 Incinerator consisted of Building 146 and three oil-fired rotary kiln incinerators with fuel storage tanks. Building 146 is still in existence; it was previously used as an explosive fill and pressure washout facility. The incinerators and fuel storage tanks have been removed. During operation, incinerator ash was stored in waste piles adjacent to the incinerator. These ash piles have also been removed. In previous investigations, sumps receiving roof drain waters and process waters were found to contain trichloroetheylene (TCE). These sumps discharged into ditches. After this discovery, a remedial action was conducted in which an air stripper was used to remove VOCs from the sumps and the roof drain waters were rerouted to a separate line discharging into a ditch. After TCE concentrations in the sump were reduced to acceptable levels, the sump drains were rerouted to the sanitary sewerage system.

The Pyrotechnic Test Area, which is still active, has been used for the functional testing of flares, signals, and other marking devices for over 40 years. This SWMU consists of three distinct areas – the Ordnance

Test Area, Annex, and Rocket Range. No analytical data are available for the Pyrotechnic Test Area. No remedial activities have been conducted at the Pyrotechnic Test Area.

Additional details on historical site operations are provided in Section 1.3 of this QAPP.

PROJECT PROBLEM STATEMENT

Because of known or suspected operationally-related chemical releases at SWMUs 12, 13, 16, and 19 (explained below), human and ecological receptors could be exposed to unacceptable health risks. The health risks are expected to be confined primarily to aqueous and solid media because only minimal airborne release pathways (e.g., occasional minor resuspension of dust or release of volatile chemicals) are anticipated.

The degree of risk to a human or ecological receptor is based on the nature of contamination and the frequency, duration, and nature of exposure to contaminants. Consequently, it is important to understand where receptors could be exposed to the contaminants. This requires that the extent of contamination be established in those SWMUs identified as exhibiting unacceptable levels of contamination. In this context, extent will be established relative to numerical risk-based criteria. The initial interest in SWMU 19 will be to determine whether any releases that may have occurred at that SWMU are great enough to warrant further investigation. If further investigation is warranted, the extent of contamination will be established. A risk evaluation must be conducted for human and ecological receptors in contaminated areas of SWMUs 12, 13, 16, and 19 to determine whether risks posed by exposure of those receptors to site contaminants are unacceptable. Plausible land use scenarios must be considered when identifying the receptors that could be at risk.

1.1 INTRODUCTION

NSWC Crane is a U.S. Navy installation located within U.S. EPA, Region 5. Tetra Tech NUS, Inc. (TtNUS) has prepared this QAPP on behalf of the U.S. Navy Southern Division Naval Facilities Engineering Command (SOUTHDIV) and NSWC Crane to comply with U.S. EPA Region 5 requirements. Those requirements and guidance, described in Section 1.1.3, govern all aspects of RCRA environmental investigations. In accordance with those requirements, project planning followed the U.S. EPA data quality objectives (DQO) process (U.S. EPA, 1999a). That process requires explicit statements of the problem to be solved, the spatial and temporal boundaries related to the problem, the measurements to be made in solving the problem and, when applicable, quantitative specifications of the tolerances for

making decision errors. The process culminates in a specification of decision rules and in a sampling and analysis plan designed to solve the stated problem.

This QAPP presents the project organization, objectives, planned activities, and specific quality assurance/quality control (QA/QC) procedures associated with sample collection and analysis for the investigation. Specific protocols for sample collection, sample handling and storage, chain of custody, chemical analyses, and data evaluation and assessment are described. These protocols are specified to assure that the data generated during this investigation are of the expected quality necessary to support project objectives.

This QAPP was prepared under Contract Number N62467-94-D-0888, Contract Task Order (CTO) Number 0166.

1.1.1 Project Objectives and Decision Statement

Past sampling and analyses, although limited, identified the presence of select contaminants at concentrations greater than proposed risk-based target levels (RBTLs) at SWMUs 12, 13, and 16. RBTLs are further discussed in Section 1.4.1.2. Previous sampling has not been adequate to delineate the extent of contamination nor has it been sufficient to support a risk assessment. This investigation is designed to further delineate the nature and extent of contamination in one or more of surface water, ground water, soil, and/or sediment, depending on the SWMU. It is also designed to provide information to implement a baseline human health risk assessment and a screening-level ecological risk assessment, including Navy Step 3A (see Section 1.4.3). Because of these two general objectives, two decision statements have been developed for this project, each of which applies to multiple environmental media. The decision statements that will facilitate attainment of the project objectives for these SWMUs are as follow:

- From ground water, surface water, soil, and sediment data, determine the nature and extent of chemical contaminant concentrations that exceed applicable screening levels in each environmental medium (surface soil, subsurface soil, sediment, ground water, surface water, and spring water). If concentrations exceeding screening levels are found, proceed to risk assessments; otherwise, take no further action.

- Determine whether contamination associated with SWMUs 12, 13, 16, and 19 poses an unacceptable risk to human or ecological receptors through the use of a baseline human health risk assessment and a screening-level ecological risk assessment. If human health risks are unacceptable, consider

implementing a corrective measures study (CMS) to evaluate options for reducing the risk to acceptable levels. If ecological risks are unacceptable, consider conducting a baseline ecological risk assessment or a CMS to reduce risk to acceptable levels. If risks are acceptable, take no further action.

Because no data are available to establish whether contaminants are present at concentrations that exceed risk-based screening levels, the investigation of SWMU 19 will begin with contaminant screening against applicable screening criteria. If warranted, this will be followed by further investigations using the two decision statements presented above. The initial decision statement applicable to SWMU 19 is as follows:

If any contaminant concentrations in soils at a particular device-testing location exceed the applicable screening criteria, conduct further investigations in accordance with the two decision statements presented above. If no contaminants are found to exceed applicable screening criteria, take no further action.

1.1.2 Project Status/Phase

SWMUs 12, 13, 16, and 19 have been investigated previously and the investigations are documented. A summary of past investigations including historical data, is presented in Section 1.3.6. Those assessments variously included sampling and analyses or a review of sampling and analyses results for surface waters, NPDES outfalls or related waters, sediments, ash piles, and soils. The environmental monitoring reports provide tabulations of potential contaminants identified at the time of the related investigations (Halliburton NUS, 1992a and b). The available data were evaluated as part of the project planning process for this investigation to establish whether additional sampling is warranted for particular environmental media and from where the samples should be collected.

At least one round of sampling is expected for this investigation for each of the four SWMUs, with additional sampling rounds possible to determine the extent of contamination or to better evaluate risk to humans or ecological receptors. The need for additional sampling rounds at a given SWMU will depend on whether data gaps exist after any given sampling round. The strategy for all sampling rounds is similar when establishing the extent of contamination. Chemical concentrations will be compared to appropriate RBTLs and background (or upgradient/upstream) concentrations to identify chemicals of potential concern (COPCs). The spatial regions in soils and ground water over which the COPC concentrations are greater than acceptable human health risk levels will be bounded. If available data is not adequate to bound contamination below risk-based levels, an evaluation will be conducted to

determine whether additional samples must be collected to complete the risk assessment and RFI process. Appropriate risk-based concentrations and background (upstream/upgradient) concentrations are described in Sections 1.1.3 and 1.4.1, 1.4.2, and 1.4.3. For surface water and sediment, establishing the nature and extent of contamination will depend on the availability of surface water and sediment, because water may be ephemeral.

The data analyses and approach to reconciling data with project objectives are described in Section 8.3.1 and throughout this document. Section 3 presents the sampling plan and analyses design and rationale for the number of soil, surface water, ground water, and sediment samples for this project.

1.1.3 QAPP Preparation Guidelines and Requirements

This QAPP has been prepared in accordance with the U.S. EPA Region 5 QAPP Policy (U.S. EPA, 1998a), which includes instructions for preparing RCRA Subtitle C corrective action investigations including RFIs. The example RCRA QAPP, included in the policy (U.S. EPA, 1998a), was followed. For establishing the QA/QC requirements specified in this QAPP, the Naval Facilities Engineering Service Center (NFESC) guidance document entitled "Navy Installation Restoration Laboratory Quality Assurance Guide" (NFESC, 1996) was followed. The NFESC guidance specifies criteria for acceptable laboratory performance and monitoring of that performance. U.S. EPA human health risk-based screening levels (RBSLs) and ecological data quality levels (EDQLs) were considered in developing this QAPP, especially when selecting sampling and analytical methods to measure target analyte concentrations in the media of interest. All QA/QC procedures are structured in accordance with applicable U.S. EPA Region 5 requirements, regulations, guidance, and technical standards.

Additional guidance regarding development of this QAPP was obtained through a pre-pre-QAPP meeting held on July 11, 2001 via conference call. Representatives from U.S. EPA Region 5, SOUTHDIV, NSWC Crane, Indiana Department of Environmental Management (IDEM), and TtNUS participated in the pre-pre-QAPP conference call to introduce and discuss technical issues associated with QAPP preparation. Concerns about the scope of the investigation were discussed and previous agreements concerning investigative approaches were reaffirmed. This teleconference was followed by an additional pre-QAPP teleconference held on September 17, 2001. At this time, the analyte lists were discussed, and the overall technical approach was agreed upon. U.S. EPA Region 5 provided written comments via electronic mail on September 18, 2001. Those comments have been addressed in this document.

Comparability of soils inorganic chemical data between this investigation and the Base-wide Background Soil Investigation for NSWC Crane (Tetra Tech NUS, 2001) is important to interpreting the data and

making background comparisons. Therefore, soil samples will be treated in a manner similar to the manner in which samples were collected for the background investigation. Data comparability is discussed further in other sections of this QAPP.

1.1.4 Organization of the QAPP

This QAPP follows the U.S. EPA Region 5 model QAPP format with minor exceptions (U.S. EPA, 1998a). An effort has been made to ensure that the flow of information from one section to another is logical, while adhering to U.S. EPA Region 5 requirements. The Table of Contents provides an overview of the document organization. Tables and figures are placed at the end of each section in which they are first referenced.

1.2 SITE/FACILITY DESCRIPTION

This section is a presentation of background information, general site characteristics of the NSWC Crane facility, and physical site characteristics specific to the four SWMUs, that are the focus of this investigation. This section contains information on topics including site location, facility size, borders, natural and man-made features, climatology, topography, local hydrology and hydrogeology, surrounding land use, and ecological communities and habitats.

1.2.1 Location and Description

NSWC Crane is located in a rural, sparsely populated region of south-central Indiana, approximately 75 miles southwest of Indianapolis, 60 miles northwest of Louisville, Kentucky, and immediately east of Burns City and Crane Village, Indiana. A location map of the NSWC Crane facility is provided as Figure 1-1. NSWC Crane encompasses approximately 62,463 acres or approximately 98 square miles of the northern portion of Martin County and smaller portions of Greene, Daviess, and Lawrence Counties.

The four SWMUs are located within the Boggs and Turkey Creek Drainage Basin, which is one of five drainage basins that carry surface water off the installation and eventually drain into the East Fork of the White River and then to the Wabash River to the southwest (Figures 1-2 and 1-3). Site features for SWMUs are included in Figures 1-4, 1-5, 1-6, 1-7, and 1-8.

1.2.2 Land Use Classification

The economic base of communities surrounding NSWC Crane is in transition from agriculture, mining, and quarrying to manufacturing and service industries. The patterns of settlement, population statistics,

and median income are similar throughout the region (TtNUS, 2000b). Because most of the region is covered by vegetation, the area is classified as rural (TtNUS, 2000b).

There is no state or local planning within the vicinity of NSWC Crane. The only zoning and land use regulations are in the municipalities in the region. None of the municipalities are close enough to impact NSWC Crane. None of the areas adjacent to NSWC Crane are zoned, and zoning is not anticipated in the near future. No known land use or community actions are being considered or proposed at this time (TtNUS, 2000).

1.2.3 Climatology and Meteorology

NSWC Crane is located in a warm, temperate climatic zone. In general, the summers are warm and humid, and winters are mild with occasional short cold periods. The temperature ranges from an average maximum July temperature of 89°F to an average minimum January temperature of 26°F. Precipitation is fairly evenly distributed throughout the calendar year; the maximum precipitation occurs during the spring and early summer. The average annual precipitation at the facility is 44 inches and consists of 42 inches of rain and 15 inches of snow. The average humidity ranges from 40 to 90 percent in summer and 60 to 90 percent in winter. Long-term climatological records for the area indicate that the monthly prevailing wind direction is from the southwest from April through December and from the northwest during January through March [National Oceanic and Atmospheric Administration (NOAA), 1988]. The annual prevailing wind direction for the region is from the southwest, and the annual average wind speed for the area is about 9.6 miles per hour. Figure 1-9 is a wind rose summarizing the mean wind direction and wind speed distribution for the Indianapolis International Airport over a 5-year period (1985-1989).

1.2.4 Physiography, Topography and Ground Cover

NSWC Crane is located in the unglaciated area of the Crawford Uplands Physiographic Province. This province is a rugged, highly vegetated, dissected plateau bounded by the Mitchell Plain Physiographic Province to the east and the Wabash Lowland Physiographic Province to the west (Murphy and Wade, 1995). The Mitchell Plain is a low dissected limestone plateau characterized by sinkholes and karst topographic features. The boundary between the Crawford Upland and the Mitchell Plain is marked by the highly irregular, eastern-facing Chester Escarpment. Springs, caverns, caves, and other solution weathering features can be found along this escarpment and on the eastern edge of the NSWC Crane facility. The boundary between the Crawford Upland and the Wabash Lowland near the western boundary of NSWC Crane is gradual (Murphy and Wade, 1995). The terrain is predominantly rolling with moderately incised stream valleys throughout and occasional flat areas in the central and northern portions

of NSWC Crane. The elevations across NSWC Crane range from about 500 feet above mean sea level (amsl) to about 850 feet amsl. Topographic relief in the Crawford Upland generally ranges from 100 to 350 feet. Greater relief exists in the eastern part of NSWC Crane near the Chester Escarpment (Murphy and Wade, 1995).

Three of the sites, Mine Fills A and B and the Cast High Explosives Fill, are located in the upland areas of the facility. The Pyrotechnic Test Area lies in lower elevations within the floodplain of Boggs Creek.

Elevations at the MFA range from approximately 745 feet amsl at the northernmost corner of the site to approximately 725 feet amsl along the western edge of the site. Surface drainage is routed through storm sewers and ditches that ultimately discharge into larger drainageways located along the eastern and western borders of the site. These drainageways ultimately flow into Turkey Creek.

Elevations at the MFB range from approximately 715 feet amsl in the northeast to approximately 650 feet amsl in the southwestern boundary of the site. Surface drainage is similar to that in MFA and is routed through storm sewers and ditches located along the northwestern and southeastern borders of the site. The ditches lead to larger drainageways that flow into Boggs Creek.

The Cast High Explosives Fill/B-146 Incinerator is approximately level, with an elevation of approximately 760 feet amsl. Surface drainage is toward the west, south, and east along storm sewers and ditches that ultimately flow into Turkey Creek.

The Pyrotechnic Test Area (Ordnance Test Area, Rocket Range, and Annex) lies within the floodplain of Boggs Creek. The elevation of the Pyrotechnic Test Area is less than 500 feet amsl, and approximately 85 to 90 percent of the SWMU is reported to lie within the 100-year floodplain of Boggs Creek (Halliburton NUS, 1992c). Surface drainage is along ditches that are routed to Boggs Creek, which bisects the Ordnance Test Area into eastern and western halves and is located adjacent to the Rocket Range and Annex.

The SWMU areas consist of buildings with either paved or gravel access roads and associated parking and staging areas. Grassy areas surround each of the SWMUs, with the exception of the Pyrotechnic Test Area, which is essentially devoid of vegetation in the treatment areas. Wooded areas surround each SWMU beyond the limits of the grassy areas.

1.2.5 Geology and Stratigraphy

The unconsolidated overburden deposits at NWSC Crane generally range from 0 feet to 65 feet thick (Nohrstedt et al., 1998). These deposits generally consist of two types: Quaternary and Pleistocene age alluvial and colluvial deposits near the floodplains of streams and unconsolidated residual soil and loess on sides and tops of ridges. The United States Department of Agriculture (USDA) has classified soils of Martin County (McElrath, 1988). Residual soils on or near the tops of ridges are generally classified as Zanesville or Wellston silt loams. These residual soils are characterized as well drained to moderately drained. They have a brown organic silt loam at the surface (typically about 8 inches thick), which is underlain by 42 to 48 inches of mottled tan, gray, and yellow clay with varying percentages of sand and silt. Occasionally, a clay hardpan occurs between 25 and 32 inches below ground surface (bgs).

Bedrock underlying the Crane facility consists of sedimentary rocks from the Lower Pennsylvanian age Mansfield Formation (Raccoon Creek Group) and the Upper Mississippian age Stephensport and West Baden Groups (Figure 1-10). The Mansfield Formation (uppermost bedrock) consists of alternating beds of shales (e.g., black shale and carbonaceous shale), sandstone, mudstone, siltstone, and thin discontinuous coal units and is typically about 110 feet thick or more (USACE, 1991). The Stephensport Group includes a number of sandstone and limestone formations, including the Big Clifty Sandstone and the Beech Creek Limestone. The Stephensport Group is generally 120 to 190 feet thick. The underlying West Baden Group also consists of limestone, shale, and sandstone units and is generally 70 to 150 feet thick.

The Pennsylvanian Raccoon Creek Group occupies the higher elevations of NSWC Crane (Figure 1-11), which includes the areas encompassing MFA, MFB, and the Cast High Explosives Fill/B146 Incinerator. Less than 10 feet of soil is reported to overlie the bedrock at these three SWMUs. Within the valley floors of NSWC Crane where the Pyrotechnic Test Area is located are alluvial (stream-deposited) and colluvial (slope debris) soils of varying thickness that overlie bedrock of the Mississippian Stephensport Group (Halliburton NUS, 1992).

Site-specific geologic information is unavailable for MFA, MFB, and the Pyrotechnic Test Area due to the absence of boring data.

The United States Army Corp of Engineers (USACE) installed monitoring wells at the Cast High Explosives Fill/B-146 Incinerator. Three to 6 feet of clayey soil overlies shale and sandstone of the Pennsylvanian Raccoon Creek Group. The shale thickness is reported to vary from 10 to 20 feet to alternating or interlayered shale and sandstone beds less than 1 inch thick. Sandstone units are reported

to be as thick as 10 to 20 feet when not interlayered with shales. Coal seams as thick as 4 feet were also encountered (Halliburton NUS, 1993).

1.2.6 Hydrology and Hydrogeology

The surface drainage at NSWC Crane has formed a dense, dendritic pattern throughout the installation. Most of the major streams flow in a general southward or southwestward direction. Seven primary creeks in five drainage basins carry surface water off the installation, where they eventually drain into the east fork of the White River and then to the Wabash River to the southwest. Figure 1-2 shows the basins and drainages of NSWC Crane. The four SWMUs lie within the Boggs and Turkey Creeks Drainage Basins, which discharge into the east fork of the White River, ultimately discharging into the Wabash River. The Boggs and Turkey Creeks drainage basins drain roughly 50 percent of NSWC Crane.

Ground water in the unglaciated southwestern portion of Indiana is generally contained in fractures and joint openings of limestone and sandstone aquifers. Aquifers are generally isolated from one another vertically by less permeable shale and siltstone units. Ground water enters the aquifers as infiltration through outcrops and flows by gravity down the dip of the strata or locally in directions controlled by the potentiometric gradients.

The Pyrotechnic Test Area is located in a topographically (and stratigraphically) lower elevation of NSWC Crane. The SWMU is located in a floodplain, and is underlain by alluvium/colluvium and the Upper Mississippian bedrock. The shallow ground water beneath the SWMU exists in the alluvium/colluvium, as well as deeper in the bedrock. Four bedrock formations comprise three aquifers of interest, the Golconda-Haney limestone (the upper aquifer), the Big Clifty sandstone/Beech Creek limestone (the middle aquifer), and the Beaver Bend limestone (the lower aquifer). The Golconda-Haney and Big Clifty/Beech Creek aquifers are interconnected, forming the uppermost aquifer.

Ground water exists in the Lower Pennsylvanian Mansfield Formation (Raccoon Creek Group) bedrock in the higher elevations of NSWC Crane, which includes MFA, MFB, and the Cast High Explosives Fill / B146 Incinerator. Ground water will most likely not be encountered in the overlying unconsolidated material at higher elevations, since that material exists above the water table. The bedrock, which includes shale and sandstone, can be alternating and interlayered in relatively thin beds; or can be thicker where not alternating or interlayered. Coal seams may also be present in the Lower Pennsylvanian bedrock. In general, ground water will travel through the individual pore spaces of coarser materials such as sandstone, or more likely, through the fracture and joint systems in the bedrock. Ground water in the

bedrock will flow through this system and along less permeable units such as shale bedding planes, which can act as a vertical barrier to downward migration.

Three shale formations beneath are important aquitards (deterrents to vertical movement of ground water and contaminants). The Indiana Springs shale underlies the Golconda-Haney limestone and is 20 feet thick. The Elwren shale is 20 feet thick and occurs at the base of the Beech Creek limestone. The Sample Formation is a 40- to 45-feet-thick black shale below the Elwren.

Site-specific hydrogeologic information is unavailable for MFA,MFB, and the Pyrotechnic Test Area due to the absence of boring data. However, shallow ground water flow patterns are expected to mimic topography, where highest ground water elevations are typically found along ridge crests and ground water flow is toward the major stream or tributary valleys. Recharge to the shallow ground water system occurs over most of the uplands and sideslopes. Ground water moves downward and then laterally, where it discharges to surface water in the deeper stream valleys.

The USACE has installed monitoring wells at SWMU 16. Ground water encountered at depths ranging from 15 to 38 feet beneath the ground surface, flows toward the south at a gradient of 0.048. Potentiometric surface contours from a previous report (Halliburton NUS, 1992b) are included in Figure 1-6.

1.3 FACILITY HISTORY AND BACKGROUND

1.3.1 Facility Operational History

NSWC Crane provides materiel, technical, and logistical support to the Navy for equipment, weapons systems, and expendable and non-expendable ordnance items. Early in 1940, Congress passed the first supplemental National Defense Appropriation Act. This act provided $5 million for new inland ammunition production facilities, $3 million of which was earmarked to build a Navy ammunition depot at Burns City on the site of the White River Project. Factors weighing in favor of the Burns City site were a remote location that was free of congested areas, hilly terrain ideal for magazine construction and camouflage, the proximity of Lake Greenwood, which could supply water for the facility, and the distance from the eastern seaboard, which minimized the danger of enemy attack.

In 1940, Congress authorized construction of a Naval Ammunition Depot (NAD) in southern Indiana, and in late 1941 the Burns City Ammunition Depot was commissioned. In 1943, NAD Burns City was renamed NAD Crane, and the town of Crane was built to house the rapidly growing number of civil

service employees. NAD Crane's overall mission was to load, prepare, renovate, receive, store, and issue ammunition to the fleet.

During World War II (WWII), NAD Crane's mission expanded to include pyrotechnics production, mine filling, rocket assembly, field storage, torpedo storage, and ordnance spare parts and mobile equipment storage. After WWII, NAD Crane supplied ammunition to the fleet during the Korean and Vietnam conflicts. During the 1950s, several new departments were created, the Ammunition Loading and Production Engineering Center (ALPEC) was transferred to Crane, and the central Ammunition Supply Control Office (ASCO) was established. During the Southeast Asia crisis, the number of full-time employees at NAD Crane grew to 6,800.

In 1975, NAD Crane was renamed the Naval Weapons Support Center (NWSC) to reflect the facility's growing involvement in high-technology weapons systems. In 1977, the Secretary of Defense combined all conventional ammunition acquisition under the responsibility of a single service. The ammunition production and storage function was given to the Army, and the Crane Army Ammunition Activity (CAAA) was established as a Crane tenant to accomplish this task for Naval ammunition. Other functions remain under the Navy, and the Navy retains ownership of all real estate and facilities at Crane. Responsibility for overall station safety, security, and environmental protection remains with the Commanding Officer, NWSC. In 1992, based on changing missions and alignment, the facility name was changed to the NSWC Crane.

Until the late 1970s and early 1980s, a variety of liquid wastes from both ordnance and non-ordnance activities were disposed on site without treatment. For example, ordnance-contaminated wastewaters were discharged into unlined ponds or into storm drains; wastewaters generated from cleaning operations were discharged into an unlined pond; oily waters, battery acids, paint thinner, acetone, boiler blowdown, and solvents were dumped behind buildings or into ravines; and waste hydraulic oil was spread on dirt roads. Since that time, wastewater treatment plants have been built, wastewater streams have been connected to the sanitary sewer, and oils and solvent have been collected, segregated, and either recycled, sold, or properly disposed.

Air pollution control devices were not installed until the late 1970s. Therefore, ventilation systems at bomb-loading facilities and the B146 incinerators had no scrubber systems until the 1970s. Waste management activities included the burning of solid waste and subsequent disposal of ash in unlined pits or gullies. Also, dye and dye-contaminated materials have been disposed in on-site trenches.

1.3.2 SWMU 12, Mine Fill A Operational History

All descriptions in this section correspond to Figure 1-4.

MFA, approximately 63 acres in size, can be divided into two halves along a central east-west line. The manufacturing and processing operations and facilities in each half are nearly identical. MFA began operations when NSWC Crane was commissioned in December 1941. MFA continued to produce ordnance until 1975. Production was resumed in 1980 for a short period of time. The mine fill is active today.

MFA has historically been used to manufacture mines, depth charges, rocket heads, aerial bombs, and projectiles. Most recently, MFA has been used in the production of 2,000-pound aerial bombs. In addition to production, demilitarization activities take place at Buildings 151, 155, and 160.

Building 151 has been used for demilitarization of Rockeye Cluster bombs. In the demilitarization process, operators remove the Rockeye bomblet from the cluster bomb. The bomblets and other explosive devices were packaged for thermal treatment at the Crane Demolition Range. All other materials were recycled through the Defense Reutilization and Marketing Office. No emissions or wastewaters were generated from this demilitarization process.

Bomb production began with inert preparation of bomb casings. Inert preparation took place in Building 155, the Empty Mine Storage Building. Bomb casings were received from the supplier and stored prior to production in Building 155. Casings selected for production were first inspected for defects and prepared for hot melt application in Building 155. Preparation included removing all plugs and shipping features from the bomb shell. Next, the interior of the bomb shell was cleaned with solvents to remove materials that may inhibit adherence of the hot melt mix to the interior of the bomb. The bomb shell was then moved to the hot melt fill station of the process control area in Building 155. At this location, the hot melt, a bituminous asphalt-type compound, was applied to the interior of the bomb and allowed to drain. The primary components of this coating were N,N-ethylene *bis*-stearamide (7%), bituminous asphalt (74.4%), and microcrystalline wax (18.6%). Following application of the hot melt, the bomb casing was moved to the grit blast chamber, where the exterior of the bomb casing was cleaned with solvents to remove excess hot melt compounds. If necessary, the bomb casing was then grit blasted to prepare the metal for painting. Hundreds to thousands of gallons of coatings were used annually. Hundreds of gallons of methyl ethyl ketone (MEK), toluene, and naphtha were used annually for cleaning. There are no known release pathways for organic solvents to have infiltrated environmental media.

After preparation, some bomb casings were moved to the painting booths in Building 155 and painted. Four paint booths are used in this process. The chemicals that were used included intumescent and polyurethane coatings, containing between five and 20 percent toluene by weight, and an enamel coating containing 37 percent naphtha by weight.

Some bombs were covered with a thermal coating called AVCO, which is known to have previously been manufactured to contain asbestos. AVCO no longer contains asbestos. After painting and any additional touch-up or detail work, the bomb casing was allowed to cure. Once the prepared and painted bomb casing had cured, it was loaded onto pouring cars and moved to the loading process area or sent to storage.

Building 155 is currently used as a renovation facility for explosives-loaded bombs and demilitarized gas generators. Building 155 contains a paint booth that has been modified to contain a waterjet system. This waterjet system is used to remove thermal coating systems from explosives-loaded bombs (500, 1000, and 2000# bombs) and asbestos liners from gas generators that have been static fired to remove any reactive materials. The wastewater and residues from the process are collected into the paint booth sump. Building 155 also is used to coat bombs in any of the other three paint booths in the facility. Operators use both conventional primers and topcoats, along with thermal coatings (AVCO). The newer AVCO mixtures do not contain asbestos.

Explosives preparation and bomb casing preparation were conducted simultaneously during bomb production runs. The following steps detail the procedures followed in explosives preparation. First, explosives were brought in by rail car to the explosives receiving building, Building 154, which was used as a temporary magazine. Explosives were taken from Building 154 to the box-emptying building, Building 153. Boxes of explosives were emptied and the explosives were sifted and scanned with magnets to remove any metallic fragments. Used explosive shipping containers were placed on conveyors at the ends of the box emptying building that transport them to one of two storage bins (box house) adjacent to the box emptying building. The conveyors were covered but the slotted conveyor belt provided a pathway for explosives debris to fall to the ground. Discarded containers remained in these storage bins (Buildings 2193 and 2195) until they were removed for disposal in areas outside of the Mine Fill.

After the explosives were sifted and scanned for metals, they were poured into covered containers and sent by conveyor to the powdered metal storage building (Building 2714) where specified quantities of metals (i.e., aluminum or other materials) were added as required for the explosive being manufactured. After ingredients were added and the cover was replaced, the containers continued to move along the

conveyor line to the upper level of the bomb-mine fill building (Building 152). In the upper level of Building 152, containers carrying explosive ingredients were emptied into a melting kettle. The ingredients in the kettle were heated to a molten state at a temperature range of 190 to 203 degrees Fahrenheit.

Prepared bomb casings were transported from the preparation area or storage area by rail car to the lower level of Building 152, the mine-bomb fill building. Molten explosives were poured from the upper level of Building 152 into the empty bomb casings below. After filling, bombs were transported by rail to the cooling and temporary storage area (Building 151). After cooling, void space left in the cooling explosive was filled and the bomb was sealed. After sealing, bombs underwent final quality assurance testing and inspection. Repairs, if necessary, were done before the bombs were shipped to storage or into service.

Demilitarization of warheads was accomplished by a steam-out and flaking out process in Building 160. The flaking out process was used to reclaim trinitrotoluene (TNT) from mines or depth charges. The steam-out process was used to remove H-6, HBX, Tritonal, and Composition B from various munitions. A new tetrytol (TNT and Tetryl) steam-out operation started on May 30, 1992. Two different methods of demilitarization were conducted at NSWC Crane. One was a steam or water-washout and the other was incineration. The washout method utilizes a stainless-steel probe connected to a steam line. The probe was inserted into the warhead to melt out the contents. Melted ammonium nitrate powder was reportedly turned into pellets and sold as fertilizer (Halliburton NUS, 1992a). Remaining explosive debris was then steamed out of the casing and the contents were sluiced to a catch pan. The water was drained from the catch pan to an aluminum holding pan. This aluminum pan was emptied several times per shift into a floor drain from which the water flowed into a portable baffled tank. Overflow from the baffled tank then flowed via floor drains to a pit outside the building, which has since been removed. The TNT left in the catch pan was placed in the TNT flaker, where it was packaged for shipment to a storage magazine. The residual TNT solids recovered in the aluminum pan and baffled tank were collected and disposed at the open burning grounds or demolition area. Other warheads were demilitarized in a similar manner, and the steamed-out contents were sluiced to a kettle where the explosives are separated from water. The explosives were sent to the open burning area or the demolition area. The water removed from the kettle flows by gravity to the same aluminum pans used in the TNT operations.

Heat used to generate the steam for the melting and steam out operations was generated by boilers that burn No. 2 fuel oil. These boilers were located in Building 150 outside MFA. The No. 2 fuel oil tank no longer exists. There are no records of any leaks from the No. 2 fuel oil tank.

Building 160 has most recently been used for the steam-out of tritonal from 750# bombs. Tritonal is a mixture of 80% TNT and 20% aluminum powder. In 1994, CAAA installed an autoclave steam-out system to replace the older steam-out racks that utilized stainless steel steam wands. Building 160 contains 20 autoclaves that are used to remove explosives from items such as bombs and projectiles, and allows for the possible reuse of the explosive filler. In the steam-out process, a bomb is loaded into an autoclave, which is then pressurized and heated (the autoclaves have a steam jacket). After the explosives in the bomb reach their melting point, the melted explosives flow out of the autoclaves into heated trace lines. The trace lines, with the aid of a pump, move the melted explosives into heated holding kettles. From the holding kettles, the explosives are transferred using vacuum-generated flow into pouring kettles. The explosives flow from the pouring kettles onto a water-cooled belt flaker that cools the melted explosives. After the explosives have cooled, they pass through a pin breaker and are placed into cardboard boxes for shipment. All of the autoclaves and associated equipment are exhausted to 6 wet scrubbers to capture any explosives before they are emitted. The building is lined with wastewater trenches, which carry any wastewaters to the explosives-contaminated wastewater treatment plant (B-3110).

Buildings 153 and 158 house explosive powders such as TNT. Aluminum powder was stored in Building 2714. Wastewater from Building 153 was previously discharged to surface drainage. The railroad lines by the box houses, the shaker rooms, and magazines were hosed down once per day. The railroad areas, where bombs and explosives were unloaded, drain to the surface water system. Engineering features to control or eliminate emissions to the air, water, and soil have been added and modified at the facility throughout the life of the facility. Prior to the installation of a particulate abatement system, powders from the operations accumulated on roof tops and were periodically washed from the roof tops. This practice, which did not include collection of the wash water, resulted in a release of explosives contaminants directly to surface soil. The wash water ran into ditches, Turkey Creek, and eventually Boggs Creek and off-station (Halliburton NUS, 1992a). One estimate of the quantities of dust released through the exhaust system was 40,000 pounds per year (Halliburton NUS, 1992a). Wastewater from the air pollution abatement scrubbers within MFA is now collected and stored in sumps.

Construction of a wastewater treatment facility at MFA was completed in 1982, but operation of the treatment facility did not start until 1986 or 1987. Building B-3110 is the wastewater treatment plant for MFA. The wastewater treatment system consists of collection sumps and three carbon adsorption columns for treatment of explosives-contaminated [e.g., TNT, HMX and cyclotrimethylenetrinitramine (RDX)] rinse waters. The sumps are checked for integrity whenever steam-out operations occur. The sumps have not shown signs of leakage. Total daily wastewater flow is estimated to be tens of thousands of gallons (Halliburton NUS, 1992a). Contaminated water is currently held in holding tanks

and tank cars (depending upon the origin) prior to treatment. After treatment, the wastewater is tested for explosives. If the explosive compound concentration is less than 2 milligrams per liter (mg/L), the water is discharged to the sanitary sewer under an NPDES permit. If the concentration is greater than 2 mg/L, the carbon columns are changed and the water is diverted through the treatment system again. Sludge is collected from the sumps and taken to the Ammunition Burning Grounds for treatment. Pumped wastewater from the sumps is transported to Building 3110 (at SWMU 12) or the Rockeye Facility for treatment.

Additional details concerning contaminants known or potentially released at MFA can be found in Halliburton NUS, 1992a. Most documented contaminant releases that occurred involved TNT only (near Buildings 151, 152, 153, 160, or 3110) or TNT plus HMX and RDX (near Buildings 160 and 3110) or an unspecified explosive powder (in the tunnel area of Building 152) (Halliburton NUS, 1992a). Note that asbestos, once a component of AVCO, is not a concern for this investigation because the asbestos fibers were entrained in the asphalt-like AVCO matrix. In that form, the asbestos is not hazardous (ATSDR, 1997).

The asphalt pad at the southern end at this SWMU has an uncertain history. It is not known why it was constructed and whether it was used although anectdotal evidence suggest that some limited flare testing may have taken place there. Contaminants likely to be present at this SWMU include explosives, semivolatile organic compounds (SVOCs), volatile organic compounds (VOCs), and metals.

On March 4, 2002, there was a 72,000-gallon release of wastewater from the scrubber building (B-3111) in the MFA. The building had not been in operation for up to a year, and all of the pumps were shut off as is normal procedure when the building is not in operation. The backflow prevention device failed, which allowed potable water to flood the building to the height of the 6-inch curb. The water then flowed out of the building, across the pavement, and into a ditch along the railroad tracks. Residual TNT from within B-3111 resulted in a small amount of TNT contamination in the water. The spilled water was sampled; and the total quantity of TNT release was well below the reportable quantity (RQ) of 10 pounds in any 24-hour period. The contaminated water contained in the building was transferred to B-3110 for treatment. This incident was reported to IDEM. An IDEM inspector visited the spill site on March 7, 2002.

1.3.3 SWMU 13, Mine Fill B Operational History

MFB operations were similar to MFA, except for larger melt kettles, additional nitrate preparation buildings (Buildings 2500 and 2501), which are located next to the box emptying buildings, and therminol boilers located near Buildings 171 and 166. The approximate 50-acre size of MFB is less than the size of MFA.

See Figure 1-5 for MFB structure locations. Only MFB operations that differ from MFA operations are presented in detail in this section. Some pollution control equipment to control air and water emissions installed at the MFA was not added at the MFB. This equipment included pinkwater treatment, wet scrubbers, and an electrostatic precipitator (ESP). This equipment controls emissions from bomb loading. The MFB does not have the capabilities for bomb loading and there are no plans to use MFB for bomb loading. Therefore, the lack of this equipment is not viewed to result in chemical releases to the environment.

As at MFA, facilities at MFB can be divided into two halves in which identical activities took place. Operations at MFB began when NSWC Crane was commissioned in December 1941. MFB has historically been used to manufacture mines, depth charges, rocket heads, aerial bombs, and projectiles. Production of explosive ordnance occurred at MFB from 1941 until 1973. MFB originally had the capability only to handle TNT. Later, this Mine Fill was modified to handle Torpex, Minol, HBX, and Amatol. During the Vietnam War, TNT, Composition B, H-6, Tritonal, and Minol were used in mine filling operations. Constituents of these explosives are RDX, TNT, aluminum powder, ammonium nitrate, and wax. Fertilizers (ammonium nitrate) were also melted into bombs. Since 1973, the facilities at MFB have been used for renovation of ordnance and equipment.

Renovation work in Building 169 includes one paint booth. The 1983 Operation Permit application contained information on these activities. The paint booth applies an enamel coating, which contains 22 percent naphtha by weight and also applies a bituminous solvent-type coating, which contains 30 percent naphtha by weight.

Loading of bullets also reportedly took place in Building 169 (Halliburton NUS, 1992a). Bullets were loaded with titanium tetrachloride and vanadium oxytrichloride or dummy loaded with sands and salts. As many as 15 gallons of titanium tetrachloride are reported to have been spilled at Building 169 and neutralized, and the residue was reportedly disposed in a sanitary landfill. These rounds are no longer loaded. Most of the vanadium oxytrichloride and titanium dioxide puff round loading equipment has been removed from this facility. Building 169 is used primarily for inert loading and painting of projectiles.

Renovation of mortars and bullets reportedly takes place in Building 165. Renovation activities include rotary grit blasting, spray-painting, propellant removal, and fuse and configuration changes. In 1998, CAAA removed the 2 paint booths from Building 165. A new building was constructed next to B-165, and an extruder was installed in this new building (B-2172). The B-165 area is now used for extrusion of C-4 blocks for various military items. In the process, C-4 is received at the building and brought to the proper temperature. The C-4 is then moved to the extruder by a bucket conveyor. The C-4 is processed

through the extruder and its associated die in a remote manner. The die cuts the C-4 into blocks, and a conveyor carries the material back to the packaging line in B-165. The blocks are inspected and packaged for shipment. This process is permitted under the Crane Title V Operating permit, although there are relatively little emissions. Building 173, formerly used to degrease mines, is no longer used. There are not records of historical spills in this area. Building 174 is used primarily for demilitarization jobs. The most recent job involved the repackaging of Mine Clearing Line Charges (MCLC). B-174 had contained a vapor degreaser, but this equipment was removed in 1993. A paint booth remains in the building, but it has not been used recently.

B-3299, which has been recently constructed, has been utilized by the Navy Ordnance Department to test small waterjet cutting systems. The building has a 6" curb and sealed floors, so no wastewater can exit the building.

Large quantities of TNT, Composition B, HBX-1, HBX-3, and H6 were reportedly collected in the sumps of MFB (Halliburton NUS, 1992a). These sumps released explosives-contaminated water directly to surface drainage channels, which flow into the Boggs Creek Watershed. Widespread explosives contamination has been reported for MFB (Halliburton NUS, 1992a).

It is assumed that polychlorinated biphenyls (PCBs) oils were heated in the therminol boilers and then transported to the melt building where the oil was used as a heat transfer medium in the melting of explosive mixtures. PCB oils are reported to have leaked or spilled from the boilers and conveyance system, thus contaminating soil (Halliburton NUS, 1992a). During the course of the remediation of explosive-contaminated soils at the MFB, soils containing PCBs were removed. No specific remediation goal for PCB was established. Confirmation sampling indicated that the remaining soils contained PCBs n excess of 10 mg/kg. Some residual PCB contamination still remains near Buildings 166 and 171 (Halliburton NUS, 1992a).

Note that asbestos, once a component of AVCO, is not a concern for this investigation because the asbestos fibers were entrained in the asphalt-like AVCO matrix. In that form, the asbestos is not hazardous (ATSDR, 1997). Contaminants likely to be present at this SWMU include explosives, SVOCs, dioxins, PCBs, and metals.

1.3.4 SWMU 16, Cast High Explosive Fill/B146 Incinerator Area Operational History

SWMU 16 is approximately 16 acres in size. Building 146 (Figure 1-6) is the largest building at SWMU 16, approximately 2/3 acre. It was an explosives fill and pressure washout facility. Most of SWMU 16 is

covered with buildings and gravel parking lots, and most grassy areas are located southeast of Building 146. The building identification numbers for SWMU 16 reflect that the site is very old (low numbers). Sumps and drains in the buildings are potential contaminant sources. Although current operations are known, it is likely that unknown operations once occurred at this site because the site is so old. Oil/water separator outfalls once discharged to swales. The outfalls have since been welded shut. Sewer lines to sumps have been rerouted to industrial sources. Down spouts have also been rerouted. There is only one sewer system at Crane and it is a sanitary sewer system.

The site is intermittently active and is used for renovation and rework and breakdown of munitions. It is sometimes used for ammunition demilitarization operations. Nearby facilities include case filling, case preparation, lunch and locker, and sewage pumping station buildings to the north and fuse and detonator magazines to the south. Magazine and storage buildings are situated east of Building 146, and magazines for fuses, detonators, and high explosives lie east of the SWMU. Both Building 145, the case filling plant, and Building 148, the small arms repacking building, housed significant quantities of explosives and smokeless powder.

In 1995, Crane Army Ammunition Activity, in conjunction with the Earth Conservancy, installed a washout system in Building 146. This system has been referred to as the WOMBAT system. The WOMBAT system is designed to use high-pressure water to wash out explosives-loaded projectiles. The washed-out explosives go through a clarifier that separates the explosives from the water. The water is re-used in the process, while the explosives are collected for packaging. This system has been through several testing phases, but has never been fully proven successful. Currently, the system is not being used. The system has containment devices to ensure that explosives-contaminated wastewaters are not released to the environment (per Doug Johnson).

Building 146 was an explosives fill and pressure washout facility with two large and one prototype oil-fed rotary kiln incinerators. Three nearby above-ground storage tanks supplied No. 2 fuel oil for the kilns. The storage tanks in the east and west bays each had an approximate capacity of 2,000 gallons, and the central storage tank feeding the prototype kiln had a 300- to 500-gallon capacity. Munitions entered the kilns at one end and were exposed to flame combustion, which resulted in the destruction of the energetics (propellants, explosives, pyrotechnics) contained within the munitions to demilitarize the munitions to demilitarize them through burning or detonation. Ash and slag residues from the incinerators were piled on the ground. The incinerators were closed in the early 1990s. A decision was made to remove the waste ash piles, and the piles were removed along with some obviously contaminated soil.

Site operations also included cast loading (melt pouring of TNT, RDX, and HBX explosives into projectiles), demilitarization and incineration of ammunitions (shotgun shells, fuses, detonators, boosters, tracers, flare candles, and smoke flares), and demilitarization and high-pressure washout. Steamout of army ammunition and rockets containing TNT and Composition B was reportedly conducted between 1954 and 1956 (Halliburton NUS, 1992b). The latter was accomplished by drilling out and recovering approximately 5 pounds of powder from each projectile. An additional 3 pounds were then washed out, collected, disposed at the burning ground. Prior to 1978, the generated wastewater was discharged through a storm drain to a ditch, with ultimate transport to Boggs Creek. After 1978, wastewaters generated at Building 146 were transported to the Rockeye Treatment Facility for treatment.

Two sumps located on the eastern and western sides of B-146 received discharges from floor drains in B-146. These sumps were designed to drain through clay tile pipes into ditches in the woods. Both sumps were expected to contain explosive-contaminated slugs. However, it was found that the sumps contained significant quantities of TCE, with the west sump having the highest concentrations. The east sump was piped to the west sump and an air stripper was installed. The clay tile discharge lines were plugged when the stripper was installed. Shortly after the air stripper was installed, it was found that stormwater draining from the roof of B-146 was flowing into the sumps and causing them to overflow. The roof drains were rerouted to separate discharge lines that discharge into ditches. The air stripper was operated until TCE levels were low enough to allow direct discharge of the sumps into the sanitary sewer system. The discharge from the sumps was rerouted to the sanitary sewer system. After the TCE concentrations were low enough to be transferred to the sanitary sewer without treatment by the air stripper, permanent pumps were installed. The TCE source was never found. A degreaser was used in B-146 during the 1980s. However, at the time that the TCE was found in the sumps, TCE was not in use in B-146 and had not been used for several years. These remedial actions were conducted during 1995 and 1996.

Major contaminants from Building 146 loading and washout activities were TNT, RDX, HBX, Composition A, Composition B, and ammonium picrate. HBX is a mixture of RDX, TNT, aluminum, and wax. Composition A contains RDX and beeswax. Composition B is composed of RDX, TNT, and wax. The primary metals of concern associated with the incinerator operations are the heavy metals lead, barium, cadmium, chromium, and mercury. Potential combustion of fuels containing PCBs and contamination from PCB oxidation products has also been reported (Halliburton NUS, 1992b). Polycyclic aromatic hydrocarbons (PAHs) and undetonated explosives have also been detected in ash from the APE-1236 incinerators. The source emission testing data, which exceeded allowable rates set under the Air Pollution Control Board of the state of Indiana, provide additional information concerning the potential particulate and gaseous releases, particularly those occurring prior to baghouse installation (Halliburton

NUS, 1992b). Initially, incinerator ash was piled near Building 146; later, ash and residue from burning and baghouse dust were collected and transferred to the storage facility. Ash from Building 146 operations has been reported to be RCRA hazardous waste (Halliburton NUS, 1992b). TCE has been detected at high concentrations at the settling basins.

Potential historical contaminant migration pathways at Building 146 include air, surface water, and ground water. Air should no longer be a migration pathway of significance because the furnaces and ash piles have been removed. Release of contaminants from surface soil or sediments to surface water and ground water is still a possibility. For example, any ash pile residue remaining in surface soils could continue to release contaminants to surface soils that then percolate into the subsurface and dissolve or be suspended in the ground water.

Site drains were terra cotta, which are known for leakage potential. Therefore, subsurface soils may be contaminated.

Ground water may be contaminated, especially because of the high concentrations of TCE found near the settling basins. The entire site is small enough that it may be considered to be a single unit with regard to ground water sampling. Contaminants likely to be present at this SWMU are explosives, SVOCs, VOCs, dioxins, and metals.

1.3.5 SWMU 19, Pyrotechnic Test Area Operational History

SWMU 19, which is approximately 240 acres in size, is also known as the Ordnance Test Area and consists of three physically separate areas where related functions are performed. Each major test area is described in the subsections below.

1.3.5.1 Ordnance Test Area (OTA)

The Ordnance Test Area (OTA), which abuts the Annex, is approximately 166 acres in size. Functional testing of flares, signals, and other marking devices has been performed at this location for about 40 years. The tested devices range from hand-held, marine, and aerial devices such as personnel distress signal flares to ground illumination signals, decoy flares, location marker flares, smoke pots, tear gas, riot and smoke grenades, and similar devices. The OTA comprises several different test locations, some of which are used for testing more than one type of device. See Figure 1-7 for OTA structure and testing locations. Testing is generally conducted on gravel pads without features to collect any residues (e.g., shrapnel) resulting from testing. However, the areas are inspected after every test, and most of the

visible debris is collected. Testing activities associated with each of the test areas are summarized in Table 1-1. In addition, herbicides are used to control vegetation in those areas, but no large-scale management of pesticides was conducted at this SWMU.

Contaminants likely to be present at the OTA are chlorates, dyes, oxidizers, fuels, inorganic salts, and other by-products of flares and smokes. In addition, jet-fuel-contaminated water is discharged onto the ground after cook-off tests, and phosphorus- and cyanide-contaminated water was discharged prior to 1989. The reactivity of phosphorus has led to the elimination of phosphorus or phosphates from the testing regime for this investigation because the elemental phosphorus should rapidly convert to phosphates upon contact with air and water.

Tear gas [alpha-chloroacetophenone (CN)] was used at the Riot Grenade Test Area at SWMU 19. However, the chemical is not expected to be of concern due to its high rate of hydrolysis in water, ease of decomposition, and low sensitivity of animals to the compound. CN has a half-life of approximately 7 minutes in water solutions with a pH of 7 at room temperature. CN can easily be inactivated by means of a water solution. Animals generally have a low sensitivity to tear gas. For example, dogs and horses can be used by police for riot control even when tear gas is being used.

The following information concerning the OTA was obtained during a teleconference among the Navy Southern Division, NSWC Crane, and TtNUS on July 11, 2001 and from the 2001 site visit by TtNUS and others:

- With some exceptions, contamination should be greatest near the test pad centers and should rapidly decrease radially outward.

- In signal test areas, the signal flares, etc. are fired vertically upward and return very close to the test pad center. Most testing is done during daylight hours, largely because of the availability of safety equipment and personnel, but sometimes the tests are conducted "after hours."

- Many of the tests emit smoke that would follow the prevailing wind direction, which is generally out of the southwest during daylight hours.

- Some signal flares and other devices are parachute suspended. For these items, the potential for longer term emission of smoke over a large area is generally much greater than land-tested devices.

Wind speed did not necessarily play a big factor in whether aerial testing would be performed; however, testing was limited when winds exceeded 15 miles per hour (mph).

- Recovery of hard debris (e.g., lead slugs) is accomplished on a visual basis. Bullets are not cleaned up in the bullet impact area. Only about 15 rounds are tested at any one time there.

- Visual observation of tested devices during and after deployment supports the estimates of contaminant distributions. For example, hand-held flares are ignited by hand and held within a few feet of the test pad centers.

- This SMWU lies in a flood plain, and distribution of contamination across the SWMU could have resulted from occasional flooding.

1.3.5.2 PTA Annex

The Pyrotechnic Test Area (PTA) Annex covers approximately 54 acres. Figure 1-7 shows important features of the PTA Annex. Rockeye bomblets and explosives testing conducted at the Annex started in 1972, but only the explosives testing continues to the present day. Rockeye bomblets were initially loaded with Octol (HMX-TNT mixture). The bomblets later contained Composition B (RDX-TNT mixture). The Annex also includes a control room, air launch building, two test chambers, two gun turrets, a metal platform and a wooden wall.

Rockeye bomblets (both live and dummy loaded) were air-fired from the air launch building into penetration test blocks set up in the first gun turret. Rockeye bomblets have been removed from the navy arsenal. The second turret is located farther west of the Annex area and was used during spin testing of Rockeye bomblets. Spin testing involves dropping a bomblet onto a steel test block placed on a steel platform at the base of the turret. A 10- to 12-foot earthen berm is located west of the Annex to help contain effects of the blasting. A 5-foot long, 4-foot high wall constructed of 4-inch by 4-inch wooden posts is located west of the earthen berm. A steel test platform and a 3-foot by 3-foot concrete pad are located west of the wooden wall. This equipment is used in testing plastic and sheet explosives.

Contaminants likely to be present in this area are explosive residues and their by-products. Metals from bomblet casings, test blocks and the guntub may also have been released at this SWMU.

1.3.5.3 PTA Rocket Range

Figure 1-8 presents important features of the PTA Rocket Range. The Rocket Range, approximately 19 acres in size, has been in use for about 40 years. Past operations involved the testing of ordnance and rocket motors. Rocket motors containing dummy loads were also tested here. A dye test tank was located at the Rocket Range during the 1950s and 1960s. Present-day testing involves smoke warheads, 5-inch rockets, and ordnance testing in test chambers (former gun turrets). The pyrotechnics are either fired down range from the launch site or are tested in one of the facilities at the Rocket Range. A sand pit is located at the impact area. A wave tank is used to test marine markers and signal flares. Most contamination at the Rocket Range should have been deposited surficially. However, some contamination at the impact area may have been released to the subsurface. Here, as in the OTA proper, contaminants may have been dispersed by flooding because the Rocket Range is in the Lake Gallimore flood plain.

The dummy loads of the rocket test should pose no chemical risks. The contaminants likely to be present in this area from pyrotechnics and other testing are similar to those for the OTA. In addition, an occasional munitions round was reportedly found during excavations in this area (Halliburton NUS, 1992a). Explosive ordnance cook-off tests are also reported to have occurred in two of the gun turret test chambers at the Rocket Range (Halliburton NUS, 1992a).

Contaminants likely to be present at this SWMU include explosives, SVOCs, VOCs, metals, cyanide, perchlorate, and nitrate.

1.3.6 Evaluation of Historical Data and Current Site Status

Several environmental investigations have been conducted at MFA (SWMU 12), MFB (SWMU 13) and the Cast High Explosive Fill/B146 (SWMU 16). Many of the historical data [contained the 1992 Environmental Monitoring Reports (EMR) for SWMUs 12 and 13 and the 1997/1998 Interim Measures Reports (IMR) for SWMU 16] were collected through programs that did not require independent data validation. Therefore, some of the data described in the following sections appear to never have undergone validation to the extent necessary for use in a risk assessment. However, data from SWMUs 12 and 13 representing soils after bioremediation has been validated [Morrison Knudson (MK) Corporation, 2000].

The following sections summarize the available historical data for each SWMU. A site investigative chronology is presented in Table 1-2.

1.3.6.1 SWMU 12, Mine Fill A

Surface water and effluent collected at various times around the MFA area revealed the presence of explosives contamination. Concentrations of TNT, RDX, and HMX in effluent and ground runoff samples collected around Buildings 152, 157, and 160 in 1972 exceeded Drinking Water Health Advisories, especially in samples collected around Buildings 152 and 157.

No explosives were detected in a ground water sample collected west of MFA in 1972. However, ground water beneath the Mine Fill may not migrate in the direction of the monitoring well. Also, in surface water samples collected in ground runoff from Building 157 in 1972, the average concentration of TNT was 0.175 mg/L and the concentration of RDX was 3.55 mg/L. In the same study, average concentrations of TNT in effluent from Building 152 and Building 160 ranged from 5.8 to 27.9 mg/L and the average concentrations of RDX and HMX in effluent from Building 152 were 35.3 and 3.7 mg/L, respectively.

In 1979, the Department of the Army performed a study to characterize wastewaters from industrial operations and to determine the biological impact of discharges upon receiving streams. Water and sediment samples were collected from two major drainage channels that carried runoff from MFA to Turkey Creek. The samples were collected from storm ditches at considerable distances from the facilities. Explosives were detected in one surface water sample at concentrations less than Drinking Water Health Advisories (the concentration of RDX was 0.05 mg/L and the concentration of HMX was 0.1 mg/L). Only HMX was detected in one sediment sample [about 1 milligrams per kilogram (mg/kg)].

Under the NPDES permit, the outfall carrying effluent from the wastewater treatment facility was regularly monitored for oil, grease, nitrate, and total explosives. Only the total explosive concentration in 1986 exceeded the discharge limit.

As part of the Initial Assessment Survey (IAS) (1985), eight soil core samples were collected around Building 153 and Building 158 and analyzed for explosives (Halliburton NUS, 1992a). The explosives concentrations in these samples were as follows: TNT ranged from 5.2 to 3,790 mg/kg, HMX ranged from 52 to 1,340 mg/kg, and RDX ranged from 349 to 15,300 mg/kg. The results indicated a "hot spot" distribution of explosives around both buildings; the highest concentrations were detected in Sample MFA-2, collected near Building 153.

In 1989, a sample of Building 151 wastewater was collected and analyzed for explosives. The sample contained 0.314 ppm of HMX, 4.2 ppm of RDX, and 124.2 ppm of TNT.

In 1997, 1998, and 1999, Morrison-Knudson Corporation (MK) and in 1999 and 2000 TolTest Incorporated (TolTest) performed sampling, excavation, on-site treatment through bioremedation (composting) of explosives-contaminated soil, and on-site backfilling of compost at MFA and MFB. The sampling and analysis program consisted of evaluation of initial characterization samples, post-excavation samples and the bioremediated soil-compost. At SWMU 12, the pre-excavation sampling was conducted around Buildings 151, 152, 153/154, 157, 158/159, and 160. The initial characterization samples were collected from 124 grid sections near these buildings. An additional 82 grid sections were added to the outer limits of the areas being characterized to delineate the horizontal extent of contamination. Samples were then collected at each of the 206 grid sections for site characterization. The initial characterization sampling at each grid location consisted of three samples, including two composite samples that were analyzed for explosives and metals and one grab sample that was analyzed for VOCs. The composites were collected at depths of 0 to 12 inches and 24 to 36 inches, and the grab sample was collected from a depth of 12 inches. The analytes selected for analysis were based on generator knowledge of process operations and the 1992 RFI (Halliburton NUS, 1992a) to ensure that solvent-contaminated soil (or other characteristic wastes) was not transported to the Bioremediation Facility for composting. The analytes for initial characterization consisted of the following:

VOCs	Explosives	Metals
methylene chloride	dinitrobenzene	aluminum
acetone	HMX	arsenic*
methyl ethyl ketone	RDX	barium
methyl isobutyl ketone	nitrobenzene	cadmium
toluene	pentaerythritoltetranitrate (PETN)	chromium
xylenes	trinitrobenzene	lead
chloroform*	tetryl	mercury
1,2-dichloroethane*	TNT	selenium
1,1-dichloroethane*	dinitrotoluenes	silver
	nitrotoluenes	
	aminodinitrotoluenes	

* = Parameter is a TCLP parameter for waste characterization (not a chemical of concern).

Based on the results of the initial characterization sampling, individual grids were selected for excavation and remediation. During excavation, field screening was used to test RDX concentrations to provide quick field screening results. Following excavation, post-excavation confirmation soil samples were

collected from the bottoms and sidewalls of each grid section and sent to a fixed-base laboratory. RDX and arsenic were the only constituents that exceeded clean-up goals in the post-excavation samples. Grid sections that were excavated were backfilled with the soil-compost. Samples were collected from each windrow of the remediated soil and analyzed for explosives only. Average concentrations of explosives in the remediated soil-compost were less than residential and/or industrial clean-up goals. The IMR for SWMU 12 evaluates the quality of the data for the bioremediation program in regard to analytical parameters such as blank contamination, duplicates, matrix spikes, etc. At least 10 percent of the data has been validated.

The following paragraphs describe soil conditions at MFA after the implementation of the bioremediation program. The information on soil conditions at MFA is detailed in the Interim Measures Report, Removal and Bioremediation of Mine Fill A Material, NSWC Crane, Crane, Indiana, [MK, October 2000] and Interim Measures Report, Removal and Bioremediation of Mine Fill A Material Addendum #1, NSWC Crane, Crane, Indiana, (MK, November 2001).

The excavation and remediation program was conducted around Buildings 151, 152, 153/154, 157, 158/159, and 160. Initial characterization samples were collected from 124 grid sections near these buildings. An additional 82 grid sections of similar sizes were added to the outer limits of the areas being characterized to delineate the horizontal extent of contamination. The analytical results from the pre-excavation and post-excavation samples are provided in Appendices D, E, and H of the IMR and IMR Addendum #1. Appendix D provides a summary of the analytical results for all pre- and post- excavation samples and indicates samples in which clean-up criteria were exceeded. Appendix E provides the initial characterization results; and Appendix F provides the post-excavation sample results. The process from initial characterization through backfilling of the bioremediated material can be followed graphically in Appendices C, G, and J of the IMR and IMR Addendum #1. Figures C1 through C6 present the results of the pre-excavation sampling by grid sections compared to clean-up criteria. Figures G1 through G4 present the results of the post-excavation samples by grid sections compared to clean-up criteria. Figures J1 through J5 indicate the grid sections into which specific windrow at bioremediated material was deposited. Based on the information provided in the IMR and IMR Addendum #1, certain areas were excavated and others were not:

<u>Areas that were not excavated</u>

- Soil concentrations in some grid sections were less than residential and/or industrial clean-up levels. Therefore, excavation and remediation of these sections were not necessary. These grid sections

are depicted in Appendices C and G of the IMR. They include the sections that were used to delineate the horizontal extent of contamination and are on the outer limits of the study areas.

- The soils in some grid sections exceeded clean-up levels but were not excavated for various reasons (e.g., the presence of utility or steam lines). This mainly applies to some grid sections around Buildings 153/154. Table 3-2 of the IMR and IMR Addendum #1 summarizes these grid sections and the reasons the soil in each grid was not excavated to clean-up levels. These grid sections are also depicted in Appendices C and G of the IMR and IMR Addendum #1. Chemical concentrations in these grid sections can be estimated by initial characterization or post-excavation data provided in Appendix E and Appendix H of the IMR and IMR Addendum #1.

Areas that were excavated and backfilled with bioremediated soil

- Soils in these grid sections were excavated because contaminant concentrations exceeded clean-up levels. The excavated areas were then backfilled with material from the bioremediation facility. The concentrations of TNT, HMX, and RDX in the remediated soil were less than industrial clean-up levels and generally less than residential clean-up levels. The average concentrations of explosives in the bio-remediated soil are provided in Table 4-4 and Appendix I of the IMR and IMR Addendum #1 for each grid section.

Based on the information in the IMR and IMR Addendum #1, the following conditions exist around specific building areas in MFA.

Buildings 153/154

Fourteen grid sections in the Building 153/154 study area (see Figure G1 of the IMR Addendum) did not meet the clean-up goals for reasons stipulated in Table 3-2 of the IMR Addendum. Twelve of these grid sections were excavated to various depths (minimum of 2 feet and maximum of 14 feet) before obstructions were encountered. The concentrations of explosives in these grid sections exceeded industrial clean-up levels in post-excavation samples located on the bottoms and sidewalls of the excavated grids. These grids sections were backfilled with bioremediated soil.

The analytical results indicate that industrial clean-up levels have been met for surface soils in all grid sections except for grid sections 27 and 28. Contaminated soil in grids sections 27 and 28 was left in place to support a steam line. According to figures provided by TolTest in September 2001, grid sections 27 and 28 have been backfilled with bioremediated soil that meets residential clean-up standard. The

bioremediated soil was placed on top of the native soil so that the concentrations in surface soil in these grid sections are less than target clean-up levels. Therefore, based on the information provided in the MFA IMR and Addendum #1, it appears that the surface soil in the area around Buildings 153/154 is now bounded by soil in which the levels of HMX, RDX, and TNT meet residential or industrial clean-up objectives.

The IMR and IMR Addendum #1 recommend no further action (NFA) for the subject areas at MFA.

Buildings 158/159

Twelve grid sections in the eastern portion of the Building 158/159 study area (see Figure G2 of the IMR) were not excavated to clean-up goals for reasons stipulated in Table 3-2 of the IMR. The concentrations of explosives in these grid sections exceeded industrial clean-up levels in post-excavation samples located on the bottoms and sidewalls of the excavated grids. These soils were excavated to various depths (minimum of 2 feet and maximum of 14 feet) and backfilled with bioremediated soil.

Soils in grid sections north and west of the study area meet residential and/or industrial clean-up levels. These sections were either not excavated because concentrations were less than clean-up levels or were excavated and backfilled with bioremediated soil.

The extent of contamination in surface soil around Buildings 158/159 appears to be well bounded by grid sections that meet clean-up levels.

Therefore, all grid sections in and around the Building 158/159 study area are less than clean-up objectives or were remediated to clean-up objectives for surface soils. The IMR recommended NFA for the designated areas around these buildings.

Building 152

One grid section (72) near Building 152 (see Figure G3 of the IMR) was not excavated to clean-up objectives because a building foundation footer was encountered. The concentrations of RDX (40.3 mg/kg) and TNT (21.4 mg/kg) in the bottom of the excavation of this grid section exceeded industrial clean-up levels in a post-excavation sample. The soils were excavated to various depths (minimum of 3 feet and maximum of 19 feet) and were backfilled with bioremediated soil-compost.

Concentrations of explosives soils in the other grid sections around Building 152 meet residential and/or industrial clean-up levels. These sections either were not excavated because concentrations were less than clean-up levels or were backfilled with bioremediated soil-compost.

The extent of contamination in surface soil around Buildings 152 appears to be well bounded by grid sections that meet clean-up levels.

Therefore, all grid sections in and around the Building 152 study area are less than clean-up objectives or were remediated to clean-up objectives for surface soils. The IMR recommended NFA for the designated areas around Building 152.

Building 157

Two grid sections (103 and 106) near Building 157 were not excavated to clean-up goals for reasons stipulated in Table 3-2 of the IMR, as shown in Figure G4 of the IMR. The concentrations of explosives in grid sections 103 and 106 exceeded industrial clean-up levels in post-excavation samples, 19.4 and 19.2 mg/kg, respectively. These soils were excavated and backfilled with bioremediated soil-compost.

Soils in the remaining grid sections are less than residential and/or industrial clean-up levels. These sections either were not excavated because concentrations were less than clean-up levels or were excavated and backfilled with bioremediated soil.

The extent of contamination around Buildings 157 appears to be well bounded by grid sections that meet clean-up levels.

Therefore, all grid sections in and around the Building 157 study area are less than clean-up objectives or were remediated to clean-up objectives. The IMR recommended NFA for the designated areas around this building.

Building 160

Five grid sections around Building 160 were evaluated in the initial characterization of SWMU 12. Chemical concentrations in four grid sections were less than residential clean-up objectives and concentrations in the other grid section were less than industrial goals. Therefore, no excavation and remediation of the soil around Building 160 were performed. The IMR recommended NFA for the area around this Building 160.

Building 151

Four grid sections around Building 151 were evaluated in the initial characterization of SWMU 12. Chemical concentrations in all grid sections were less than residential clean-up objectives. Therefore, no excavation and remediation of the soil around Building 151 were performed. The IMR recommended NFA for the area around this Building 151.

1.3.6.2 SWMU 13, Mine Fill B

As with MFA, the major sources of contamination at MFB were washdown from buildings onto the ground and exhausts vented from ventilation systems. Also, therminol boilers located near Buildings 166 and 171 leaked PCB oils. The boilers and surrounding soil were removed in 1989, but subsequent soil samples collected near the boilers indicated that PCB concentrations were greater than 10 mg/kg.

The Department of the Army selected surface water samples were collected in drains and ditches in 1972, 1978, and 1979 by the Department of the Army (Halliburton NUS, 1992a). Samples were collected adjacent to buildings and at greater distances from the buildings. TNT, RDX, and HMX were detected in most samples, and concentrations in the samples collected nearer the buildings were greater than the concentrations in the more distant samples. In 1972, surface water samples collected in drains near Buildings 166, 167, 168, 171, 174, 2501, 2502, and 2503 were found to contain TNT (10.1 to 15.4 mg/L), RDX (0.4 to 20.4 mg/L), and HMX (0.26 to 2.4 mg/L). The greatest concentrations were detected in the south drain near Buildings 171, 174, 2501, and 2503. In 1978, drainage and runoff samples from the north and south contained TNT (0.24 to 7.7 mg/L), RDX (0.865 to 16 mg/L), and HMX (0.46 mg/L); the greatest concentrations were in the south runoff. Surface water samples were collected in ditches farther from the facilities in 1979. TNT was detected in one sample at a concentration of 0.253 mg/L (central ditch). RDX was detected in the south-central and central ditches at concentrations of 0.6 and 2.7 mg/l, respectively. HMX was detected in the south-central and central ditches at concentrations of 0.3 and 0.3 mg/l, respectively. Sediment samples were collected from ditches located farther from the buildings in 1979. TNT (0.1 to 2.0 mg/kg), RDX (20 to 28 mg/kg), and HMX (3.4 to 36 mg/kg) were detected in the sediment samples. Data from these sampling events exhibited the same pattern: samples from the north ditch had no detectable explosives; samples from the central and south-central ditches had measurable concentrations of TNT, RDX, and HMX in both the aqueous and sediment fractions; and samples from the south ditch had no detectable explosives in water but contained high levels of RDX and HMX in sediment. The quality of the surface water and sediment data is not known, and the data do not appear to have been validated.

Pilot test studies were conducted from three hand-dug wells located in the vicinity of MFB in 1972 (Kent et al., 1972). Water samples were collected from these wells and only one well (W036) contained detectable levels of explosives (at concentrations exceeding risk-based Drinking Water Health Advisories). Concentrations of TNT ranged from 0.1 to 5.8 mg/L, and concentrations of RDX ranged from 0.1 to 10 mg/L. Kent et al. (1973) also reported soil sample concentrations in ditches near Buildings 172 and 2501 with concentrations of TNT and RDX at percent levels (concentrations of TNT ranged from 0.12% to 25% and RDX ranged from 0.05% to 58%). The greatest concentrations were found in the soil collected from the ditch northeast of Building 2501.

As part of the IAS for SWMU 13, eight soil samples were collected around Building 167 and Building 172 from core soil samples to a 20-inch depth in 1985 and analyzed for explosives (Halliburton NUS, 1992a). Concentrations of TNT ranged from 4.8 to 2,410 mg/kg, HMX ranged from 10.8 to 2,020 mg/kg, and concentrations of RDX ranged from 20.6 to 24,100 mg/kg; the greatest concentrations were in sample MFB-5, located near Building 172. The data indicated that the area around Building 172 was much more contaminated than the area around Building 167.

In 1985, a soil sample was collected from the area around Building 169. The sample was tested by the EP Toxicity Test for susceptibility of contaminants to leach. Concentrations of metals, methylene chloride, and PCBs were below 1990 toxicity characteristic leaching procedures (TCLP) action levels.

MFB was remediated in 1999, 2000, and 2001 as part of the bioremediation program described in Section 1.3.6.1. The types of analytes measured before and after remediation were the same as for SWMU 12 with the addition of PCBs (Arochlor 1242, 1254, 1260).

1.3.6.3 SWMU 16, Cast High Explosive Fill/B146 Incinerator

Quantities of chemicals potentially released at SWMU 16 are unknown. Analytical data for two ash pile samples collected in 1982 show that the ash from the rotary kiln furnaces was a RCRA hazardous waste. Barium, cadmium, and lead exceeded the EP Toxicity limits (Halliburton NUS, 1992b).

Surface soil and sediment samples were collected around Building 146 in 1982 and 1985. The 1982 samples exceeded EP Toxicity action levels for cadmium (1.0 mg/L) and lead (5.0 mg/L). These samples contained trace levels of explosives (RDX, to 0.013 mg/kg and HMX, 0.022 mg/kg) and high oil and grease content. One of the 1985 samples exceeded the EP Toxicity action level for lead. None of the

1985 samples contained detectable levels of explosives. However, the detection limits were relatively high (TNT = 3.6, RDX = 5, and HMX = 7.2 mg/kg).

During some rerouting of drain lines, trenches were dug through the asphalt cover and into the soil. Samples were collected from the soil/asphalt mixture, and low concentrations of PCBs were detected in the samples.

Sediment samples collected from drainageways in the north ditch where the Building 146 drains discharge (Department of the Army, 1980) contained TNT (109 to 383 mg/kg), RDX (31 to 44 mg/kg), and HMX (1.0 to 10.2 mg/kg). In 1979, water samples were collected (from the same areas as the sediment samples) near settling basins or sumps where Building 146 floor drains discharge. None of the water samples contained detectable levels of HMX. The TNT concentration was 0.11 mg/L and RDX was 0.03 mg/L. No measurable concentrations or related detrimental effects were found in Turkey Creek, the ultimate receptor of the Building 146 discharge. A 1972 water sample from Building 146 was reported (Halliburton NUS, 1992b) to contain detectable levels of HMX and RDX, but the concentrations were not provided.

Soil samples were collected from the Building 146 incinerator bays as part of closure activities. Between October and December 1989, 133 soil samples were collected from 34 locations from depths between 0 and 2 feet bgs. The samples were analyzed for metals and PCBs. In March 1990, an additional 16 samples were collected from four locations to represent background conditions. The Indiana Department of Environmental Management (IDEM) determined that QA/QC data for the PCB parameters were inadequate, and six locations were resampled from 0 to 2 feet bgs (IDEM, 1990). The analytical results for samples collected from below the incinerator bays indicated that concentrations of arsenic, cadmium, chromium, selenium, and silver exceeded background levels. Only the concentrations of selenium and silver were considered significant (Halliburton NUS, 1993), but concentrations were not provided. The PCB analytical data indicated that detectable levels of PCBs were not present in Building 146 sampling locations.

Four monitoring wells were installed near Building 146 in July 1983. Soil and ground water samples were collected and analyzed for metals and selected organic constituents. Based on a comparison of concentrations with average United States metal concentrations, the soils around Building 146 contained elevated concentrations of lead (9.3 to 58 mg/kg) and zinc (19 to 280 mg/kg). Ground water samples around Building 146 exceeded drinking water regulations for lead (0.5 to 0.2 mg/L), chromium (0.038 to 0.16 mg/L), nickel (0.16 to 0.55 mg/L), cadmium (0.006 mg/L), mercury (0.0007 to 0.0036 mg/L), and

TCE, the only organic chemical detected (2.7 to 27 mg/L). TCE was not detected in soil borings collected from 0 to 5 feet at the same locations.

1.3.6.4 SWMU 19, Pyrotechnic Test Area

Analytical data for SWMU 19 are not available. The 1992 Halliburton NUS Phase II Assessment Report states that, "Based on waste characteristics, visual evidence, operational practices, and locational characteristics, potential releases to ground water and surface water were deemed likely. Based on analytical reports and direct visual observations, releases to soil and air have occurred. RFI Phase II Release Assessments for these media were recommended." The investigation proposed in this QAPP is intended to supply sampling and analytical data for SWMU 19.

1.4 INTENDED DATA USES

This section provides a detailed description of the project target parameters and intended data uses.

All field parameter results greater than or equal to method detection limits (MDLs) will be reported. Field parameters not detected will be reported at the MDL. MDLs for field parameters are based on method or test kit capabilities and specifications. All laboratory results greater than or equal to threshold values (TVs) will be reported. Target parameters not detected will be reported at the TV. TVs for laboratory parameters are based on method capabilities and specifications. They reflect the computed method detection limit that has been adjusted to reflect what the laboratory realistically can expect to achieve on a routine basis for the environmental matrices to be analyzed. This accounts for day-to-day variations in MDLs that the laboratory has experienced in the past, plus other factors such as the recognition that MDLs were computed from data generated in matrices other than those at SWMUs 12, 13, 16, and 19.

Analytical results for analytes that are less than applicable MDLs (or TV) will be reported with a "U" flag. The "U" flag signifies that the parameter was analyzed for but was not detected at a concentration greater than or equal to the MDL (or TV). Analytical results that are between the MDL (or TV) and the reporting limit (RL) will be reported with a "J" flag. Laboratory TVs are based in part on best professional judgment and on statistical computations in accordance with 40 Code of Federal Regulations (CFR) Part 136, Appendix B. The CFR requires the MDL to be computed as the standard deviation of replicate analyses, with results multiplied by the appropriate Student's t-value. Refer to Laucks SOP LTL-1011 for a mathematical computation of instrument detection limit (IDL)/MDL. Sample-specific laboratory TVs will be computed for each sample to account for variations in the TV that are caused by factors such as sample moisture content, the size of the sample aliquot used in the analysis, and dilutions. The actual TV used

will be an estimate of the MDL achievable in actual sample matrices. This estimate will be based on the laboratory's computed MDLs and the laboratory's best estimate of the effect of actual sample matrices on these computed values. This approach will frequently result in a TV that is greater than the computed MDL but less than the practical quantitation limit (PQL). It is viewed to be a reasonable compromise between using MDLs that are unrealistically low and PQLs that reflect the quality of the concentration estimate rather than the ability to detect the presence of an analyte in a sample.

1.4.1 Project Target Parameters and Rationale for Selection

Key target parameters for SWMUs 12, 13, 16, and 19 for each environmental medium were selected based on historical activities, types of contaminants that may have been released as a result of the activity conducted, and available historical monitoring data.

SWMU 12

The SWMU 12 operational history is presented in Section 1.3.2. The primary contaminant deposition mechanisms are as follows:

- Explosives powders washed from roof tops onto surface soils
- leakage of contaminants from sumps
- direct discharge of contaminants from the wastewater treatment system drainage bypass line

These deposition mechanisms affect primarily surface soils and, in locations near sumps, subsurface soils. Contaminants in surface soils may have migrated into the subsurface. Past sampling and analyses of select soil locations demonstrate that this has occurred. Contaminated surface soil also may migrate as the result of overland flow into site drainage channels and ultimately into Turkey Creek, which is adjacent to SWMU 12. Surface water may be contaminated via contact with contaminated surface soils and sediments. Subsurface soils could release contaminants to ground water.

The chemicals or chemical mixtures potentially released at SMWU 12 fall primarily into two categories: metals and high explosives. Metals concentrations reported previously have not been cause for concern but explosives concentrations were elevated. The elevated explosives concentrations have been reduced through remediation. VOCs and SVOCs could be a concern because of potential use at the unidentified asphalt pad (UAP).

Existing analytical methodologies are sufficient to quantify these chemicals in each of the environmental matrices of interest for comparison to risk-based screening criteria and for risk assessment. Refer to Table 1-7 for specific analytical methodologies and corresponding target analyte lists. Thus, the following analytical scheme has been selected:

- Metals (surface and subsurface soils, surface water, sediments, and ground water) – Aluminum and other metals may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water. Select locations near the UAP will be targeted for soils analysis.

- SW-846 nitroaromatics/nitramines (surface water, sediments, and ground water) – These compounds may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water. Soils have been remediated to concentrations that are generally less than applicable industrial risk levels, and some soils were remediated to less than residential risk levels. Soils near the UAP and settling basins will be sampled and tested for explosives. Unless data from this investigation indicate that further evaluation of surface and subsurface soils is warranted, these media will not be investigated further.

- VOCs (subsurface soil) - VOCs may have been released to subsurface soils near the UAP.

- SVOCs (surface and subsurface soils, surface water, and sediments) - SVOCs, including polycyclic aromatic hydrocarbons (PAHs), may have been released into soils and accumulated near the UAP. The potential exists for migration toward downstream surface water conveyances.

- General water-quality parameters [dissolved oxygen, oxidation reduction potential (ORP), pH, specific conductance, temperature, turbidity, and flow rate (surface water only)] – Information on general water-quality parameters is necessary to evaluate the overall quality of Turkey Creek tributaries surface water and to provide information on contaminant masses (flow rate).

- Total organic carbon (TOC), cation exchange capacity (CEC), and pH – (soils and sediments) –This provides information for use in corroborating the absence or presence of contamination and potential bioavailability.

- Depositional environment and grain size (sediment, surface soil, and subsurface soil) – This provides information for potential use in the CMS and for comparison to background concentrations (for naturally occurring inorganics).

- Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS/SEM) (sediment) – This provides information for fate and transport of contaminants. AVS/SEM metals will be reported separately from total metals.

- Ammonia (surface water and ground water) - This parameter will be analyzed to determine the impact of ammonia in surface water and ground water. Ammonia may be leaching from bioremediated soils.

- Nitrate (ground water and surface water) - This will be tested to evaluate the impact of nitrate in ground water and surface water. Nitrate is a degradation product of certain explosives.

Table 1-3 provides the same information for SWMU 12. The specified data uses are linked to the decision statements presented in Section 1.1.1. Data used to determine the nature and extent of contamination will also be used to evaluate human health and ecological risk.

SWMU 13

The SWMU 13 operational history is presented in Section 1.3.3. The primary contaminant deposition mechanisms are as follows:

- powders washed from roof tops onto surface soils
- leakage of contaminants from sumps
- direct discharge of contaminants from the wastewater treatment system drainage bypass line
- leakage of oils containing PCBs from therminol boilers

These deposition mechanisms affect primarily surface soils in locations near sumps and subsurface soils. Contaminants in surface soils may have migrated into the subsurface. Past sampling and analyses of select soil locations demonstrate that this has occurred. Contaminated surface soil also may migrate as the result of overland flow into site drainage channels and ultimately into Turkey Creek, which is adjacent to SWMU 13. Surface water may be contaminated via contact with contaminated surface soils and sediments. Subsurface soils could release contaminants to ground water.

The chemicals or chemical mixtures potentially released at SMWU 13 generally fall into four categories: metals (aluminum), SVOCs (bitumin), VOCs (naphtha), and high explosives (TNT, H-6, HBX, Tritonal, Composition B, and Tetrytol). Recent Mine Fill B Interim Measures data indicate that VOC concentrations in soil were less than detectable levels in most cases and that the detectable concentrations were much less than residential cleanup levels. Existing analytical methodologies are sufficient to quantify these chemicals in each of the environmental matrices of interest for comparison to risk-based screening criteria and for risk assessment. Refer to Table 1-7 for specific analytical methodologies and corresponding target analyte lists. Thus, the following analytical scheme has been selected:

- Metals (sediments, surface water, and ground water) – Aluminum and other metals may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water. Past analyses of soils generally have not shown metals to be cause for concern.

- SW-846 nitroaromatics/nitramines (sediments, surface water, and ground water) - These compounds may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water. Soils have been remediated to concentrations that are generally less than applicable industrial risk levels, and some soils were remediated to less than residential risk levels. Unless data from this investigation indicate that further evaluation of surface and subsurface soils is warranted, these media will not be investigated further.

- VOCs (surface and subsurface soils and ground water) - VOCs may have been released directly to surface or subsurface soils near the therminol boilers.

- SVOCs (surface and subsurface soils, sediments, surface water, and ground water) – SVOCs, including PAHs, may have been released directly to surface or subsurface soils near the therminol boilers. Sediments will be used to establish, at least in part, the influence of surface soil runoff because they represent the accumulation of surface water runoff from a relatively large area.

- PCBs (surface and subsurface soils near the therminol boilers) - Existing evidence shows that soils near the therminol boilers are still contaminated with PCBs. These compounds are not very soluble in water so water samples will not be analyzed for PCBs.

- Dioxins and furans (surface and subsurface soils) - These chemicals may have been generated from combustion of PCB oil. The chemicals may have been released via air dispersion to surface soil with

migration into subsurface soil. These compounds are not very soluble in water so water samples will not be analyzed for dioxins and furans.

- General surface water quality parameters [dissolved oxygen, ORP, pH, specific conductance, temperature, turbidity, and flow rate (surface water only)] – Information on general water-quality parameters is necessary to evaluate the overall quality of Turkey Creek tributaries surface water and provide information on contaminant masses (flow rate).

- Ammonia (surface water and ground water) - This parameter will be analyzed to determine the impact of ammonia in surface water and ground water. Ammonia may be leaching from bioremediated soils.

- Nitrate (surface water and ground water) - This information is necessary to evaluate the impact of nitrate on ground water and surface water. Nitrates are degradation products of certain explosives.

- TOC, CEC, and pH (soils, and sediments) – This provides information for use in corroborating the absence or presence of contamination and potential bioavailability.

- Depositional environment and grain size (surface soil, subsurface soil, and sediment) – This test will provide information for potential use in the CMS and for comparison to background concentrations (for naturally occurring inorganics).

- AVS/SEM (sediment) – These tests will provide information for fate and transport. AVS/SEM metals will be reported separately from total metals.

Table 1-3 provides the same information for SWMU 13. The specified data uses are linked to the decision statements presented in Section 1.1.1. Data used to determine the nature and extent of contamination will also be used to evaluate human health and ecological risk.

SWMU 16

The SWMU 16 operational history is presented in Section 1.3.4. The primary contaminant deposition mechanisms are as follows:

- Combustion exhaust released to the atmosphere
- Discharge of wastewater to surface drainages
- Releases from waste ash piles

These deposition mechanisms affect primarily surface soils, sediments, and surface water. Combustion products would settle on surface features and could be washed into drainage channels by overland flow and wind erosion. However, air migration should no longer be a migration pathway of significance because the furnaces and ash piles have been removed. Contaminants in surface soils may have migrated into the subsurface. Past sampling and analyses of select soil locations demonstrate that this has occurred. Contaminated surface soil also may migrate as the result of overland flow into site drainage channels and ultimately into Boggs Creek, which is downgradient of SWMU 16. Surface water may be contaminated via contact with contaminated surface soils and sediments. Subsurface soils could release contaminants to ground water.

The chemicals or chemical mixtures potentially released at SMWU 16 generally fall into five categories: metals (aluminum, lead, cadmium, chromium, barium, mercury), PCBs, PCB oxidation products (dioxins and furans), VOCs, and high explosives (Compositions A and B, HBX, and TNT). Ammonimum picrate is not covered by these categories. Existing analytical methodologies are sufficient to quantify these chemicals in each of the environmental matrices of interest for comparison to risk-based screening criteria and for risk assessment. Refer to Table 1-7 for specific analytical methodologies and corresponding target analyte lists. Thus, the following analytical scheme has been selected:

- Metals (surface and subsurface soils, surface water, sediments, and ground water) – Metals may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water.

- SW-846 nitroaromatics/nitramines (surface and subsurface soils, surface water, sediments, and ground water) – These compounds may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water.

- Picrates (surface and subsurface soils, surface water, sediment, and ground water) - Releases of picrates (ammonium picrate in particular) may have affected all media because the relatively soluble picrates would exhibit good migration potential.

- VOCs (surface and subsurface soil and ground water) - Contaminants released to soils in the 1980s or to sumps may have migrated deeper to subsurface soils and ground water.

- SVOCs (surface and subsurface soils, surface water, sediments, and ground water) – SVOCs, including PAHs, may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water.

- PCBs (surface and subsurface soils) – These compounds may have been released directly to surface or subsurface soils with subsequent migration to sediments. The PCBs are not very soluble in water and, therefore, have limited migration potential.

- Dioxins and furans (surface and subsurface soils) – These compounds may have been released directly to the air and would have settled on surface soil with limited migration detected to ground water and downstream surface water channels.

- General surface water quality parameters [dissolved oxygen, ORP, pH, specific conductance, temperature, turbidity, and flow rate (surface water only)] – Information on general water-quality parameters is necessary to evaluate the overall quality of Turkey Creek tributaries surface water and provide information on contaminant masses (flow rate).

- Nitrate (ground water and surface water) - This test is to evaluate the impact of nitrates on the ground water and surface water. Nitrates are degradation products of certain explosives.

- TOC, CEC, and pH (surface and subsurface soils, and sediments) – This provides information for use in corroborating the absence or presence of contamination and potential bioavailability.

- Depositional environment and grain size (sediment, surface soil, and subsurface soil) – This test provides information for potential use in the CMS and for comparison to background concentrations (for naturally occurring inorganics).

- AVS/SEM (sediment) – This test provides information for fate and transport. AVS/SEM metals will be reported separately from total metals.

Table 1-4 provides the same information for SWMU 16. The specified data uses are linked to the decision statements presented in Section 1.1.1. Data used to determine the nature and extent of contamination will also be used to evaluate human health and ecological risk.

SWMU 19

The SWMU 19 Ordnance Test Area operational history is presented in Section 1.3.5. The primary contaminant deposition mechanism is aerial release directly to surface soils and sediments. Some contaminants are likely to have been dispersed by the wind and settled onto surface features such as surface soil and surface water and sediments. However, wind dispersal may have incurred significant dilution of the chemicals. Most contaminants are expected to be found at the greatest concentrations in surface soils near the test locations. While contaminants in surface soils may have migrated into the subsurface, determining the extent of contamination will not be necessary unless contaminant concentrations exceed screening criteria.

With a few exceptions, the chemicals or chemical mixtures potentially released at SMWU 19 generally fall into five categories: inorganic chemicals, SVOCs, VOCs, dyes, and high explosives. Existing analytical methodologies are sufficient to quantify these chemicals in each of the environmental matrices of interest for comparison to risk-based screening criteria and for risk assessment. With some modifications, all the chemicals potentially released at SMWU 19 or representative chemicals can be quantified using existing analytical methods. Refer to Table 1-7 for specific analytical methodologies and corresponding target analyte lists. Thus, the following analytical scheme has been selected:

- Metals (surface and subsurface soils, sediments, surface water, and potentially ground water) – Aluminum and other metals may have been released directly to surface or with subsequent migration to subsurface soils, sediments, surface water, and ground water.

- SW-846 nitroaromatics/nitramines (surface and subsurface soils, sediments, surface water, and potentially ground water) – These compounds may have been released directly to surface or subsurface soils with subsequent migration to sediments, surface water, and ground water.

- VOCs (surface and subsurface soils and potentially ground water) – These compounds may have been released directly to surface or subsurface soils near the cook-off testing areas with potential migration to other media.

- SVOCs (surface and subsurface soils, sediments and potentially surface water and ground water) – These compounds may have been released directly to surface soils in the vicinity of cook-off operations, with subsequent migration to other media.

- Dyes (surface soil) - These compounds may have been released directly to soil surfaces in the vicinity of the ordnance testing area.

- Perchlorate (surface and subsurface soils, sediments, surface water, and potentially ground water) – These chemicals, which are widely used in propellants and explosives, may have been released to surface soils at various locations across this SWMU with subsequent migration to other media.

- Cyanide (surface and subsurface soils and potentially ground water) – This chemical may have been released to surface soils near cook-off operations and has been detected in soils previously. If the first round of sampling shows cyanide to be present in solid media, ground water may be analyzed for cyanide.

- General surface water quality parameters [dissolved oxygen, ORP, pH, specific conductance, temperature, turbidity, total suspended solids, and flow rate (surface water only)] – Information on general water-quality parameters is necessary to evaluate the overall quality of Boggs Creek surface water and provide information on contaminant masses (flow rate).

- TOC, CEC, and pH (surface and subsurface soils, sediments) – This provides information for use in corroborating the absence or presence of contamination and potential bioavailability.

- Depositional environment and grain size (surface soil, subsurface soil, and sediment) – This test provides information for potential use in the CMS and for comparison to background concentrations (for naturally occurring inorganics).

- AVS/SEM (sediment) – This test provides information for fate and transport. AVS/SEM metals will be reported separately from total metals.

Table 1-5 provides the same information for SWMU 19. The specified data uses are linked to the decision statements presented in Section 1.1.1. Data used to determine the nature and extent of contamination will also be used to evaluate human health and ecological risk.

1.4.1.1 Field Parameters

Several field measurements will be made for this investigation. As previously noted, Tables 1-3 through 1-5 summarize all project target parameters and the associated intended data uses. Table 1-6 indicates

which measurements will be made in the field for aqueous samples and presents MDLs, as applicable, for the aqueous target parameters measured in the field.

1.4.1.2 Laboratory Parameters

Laboratory analyses will be used to estimate target analyte concentrations in ground water, surface water, surface soil, subsurface soil, and sediment. Use of target analyte data for decision making is described in Sections 1.4.2, 1.4.3, 1.4.4, and 12.4.

Part of the data use includes comparing metal concentrations in soil at the individual SMWUs where soil or sediment is contaminated to greater than soil background concentrations for naturally occurring metals. The background concentrations for soil types found at the SWMUs will be obtained from the NSWC Crane Base-Wide Background Soil Investigation (TtNUS, 2001a). This report contains concentrations of naturally occurring metals in soil types found at the NSWC Crane SWMUs, including SWMUs 12, 13, 16, and 19.

The data use also includes comparing the analytical data to RBTLs, which are action levels derived from human health and ecological risk-based screening levels (RBSLs). The RBTL is the lowest (i.e., most conservative) RBSL. Following is a list of sources for the RBSLs applicable to this project:

<u>Ground Water and Surface Water RBSLs</u>

- Federal Maximum Contaminant Levels (MCLs)
- U.S. EPA Region 9 Preliminary Remediation Goals (PRGs) for Tap Water
- IDEM Tier I Default Residential and Commercial/Industrial Ground Water Closure Levels
- U.S. EPA Region 5 Ecological Data Quality Levels (EDQLs) for Surface Water
- Federal Ambient Water Quality Criteria (AWQC) for Freshwater

<u>Soil and Sediment RBSLs</u>

- U.S. EPA Generic Soil Screening Levels (SSLs) for Ingestion, Inhalation, and Migration to Ground Water
- U.S. EPA Region 9 PRGs for Residential and Industrial Land Use
- IDEM Tier I Default Residential and Commercial/Industrial Soil Closure Levels
- U.S. EPA Region 5 EDQLs for Soil (applicable to surface soil samples only)
- U.S. EPA Region 5 EDQLs for Sediment (applicable to sediment samples only)

Tabular presentations of the RBSLs are provided in Appendix B. Table 1-7 lists the laboratory parameters, analytical methods, laboratory detection and reporting limits, and RBTLs for water (applicable to both ground water and surface water), soil, and sediment. MDLs are generally less than RLs. Measurement uncertainty is so great at concentrations less than the MDL that the presence of an analyte cannot be asserted with reasonable confidence in that concentration range. Thus, results less than the MDL represent analytes that are labeled as "non-detects." As concentration increases, the relative measurement uncertainty typically decreases up to the RL. Analyte concentrations greater than RLs are generally reported with a relatively high degree of accuracy (i.e., the reported value is within about 20 or 30 percent of the true value). The uncertainty varies from analyte to analyte and is not typically quantified for individual analytes. The uncertainty associated with results between the MDL and RL is comparatively high but also varies from analyte to analyte. This can cause problems when interpreting data, especially when comparing two values. However, the decision to report to concentrations as low as MDLs was made to provide the greatest chance for achieving the RBTLs in Table 1-7. Despite this relatively aggressive reporting convention, several RBTLs in both aqueous and solid media are still unattainable. TtNUS has worked closely with the subcontracted analytical laboratories to select and optimize analytical methods in an effort to attain, to the greatest extent possible, laboratory MDLs (or even RLs) that are less than or equal to the RBTLs. It is widely recognized that MDLs computed according to the industry standard (40 CFR Part 136, Appendix B) are determined in sample matrices that are relatively simple. As such, they do not necessarily reflect what is achievable in actual environmental sample matrices. MDLs in Table 1-7 reflect the computed MDL based on 40 CFR protocol tempered by the laboratory's experience with analyzing samples with more complex matrices than those used to determine the MDLs. Therefore, the MDLs in the tables may be greater than the computed MDLs. Analytes for which the MDL is greater than the RBTL for either aqueous or solid matrices are identified in Table 1-7 by shading.

1.4.2 Human Health Risk Assessment

A baseline human health risk assessment will be prepared to estimate risks to current or hypothetical future receptors at SWMUs 12, 13, 16, and 19. The detailed risk assessment methodology is presented in Appendix C. The methodology considers risk assessment protocols established by U.S. EPA Region 5 and the IDEM. The following components of a baseline risk assessment (BRA) are included in Appendix C:

- Data evaluation protocol (including data usability assessment; COPC selection)
- Exposure assessment (including CSM)

- Toxicity assessment
- Risk characterization
- Uncertainty analysis

1.4.3 Ecological Risk Assessment

The screening-level ecological risk assessment (SERA) will consist of the first two of eight steps required in the U.S. EPA guidance (U.S. EPA, 1997 and 1998b). Step 3A (of the Navy Policy for Conducting Ecological Risk Assessments) is the first step of the baseline ecological risk assessment (BERA) and consists of refining the list of COPCs that were retained following the SERA. Step 3A will be included as an attachment to the SERA. Steps 3B through 7 will be conducted if additional evaluations or investigations are necessary. Finally, Step 8, Risk Management, will be incorporated throughout the ecological risk assessment (ERA) process in cooperation with Region 5 regulators.

The first phase in the ERA process is the screening-level risk assessment. In this phase, conservative exposure estimates are made for grouped or individual ecological receptors, and these exposures are compared to screening levels and threshold toxicity values. The SERA includes the following considerations, which are described in Appendix D:

- Screening-level problem formulation
- Screening-level ecological effects evaluation
- Screening-level exposure estimate
- Screening-level risk calculation

1.4.4 Decision Rules

Based on site-specific factors described above, and the DQO process outputs, this section describes how the data will ultimately be used for making decisions concerning the nature and extent of chemicals and the risks to human health and ecological receptors.

The overall strategy for SWMUs 12, 13, 16, and 19 is to first screen analytical results against risk-based screening criteria to select COPCs. If COPCs are identified during screening, data assessment continues with an evaluation of the nature and extent of contamination and an evaluation of the associated human and ecological risks. Additional data collection may be necessary. This is especially true for SWMU 19 because of the limited amount of data that will be available, initially. If COPCs are identified, it is highly likely that additional sampling will be required at SWMU 19 to support a determination of the nature and

extent of contamination and to support the evaluation of risk. Additional sampling beyond the second round of sampling is less likely.

Decision rules are designed to be technically defensible and practical to implement. The decision rules below apply to all target analytes except field and geotechnical parameters, which have no bearing on COPC selection or evaluations of risk.

To select COPCs, site chemical concentrations will be compared to RBTLs and background or upgradient concentrations. For determinations of nature and extent, risk contours will be plotted. The contours will be based on cumulative risk estimates calculated for COPC concentrations at the sampled locations. At a minimum, plots representing the 1E-4 cancer risk level and a Hazard Index (HI) of 1 will be presented. Contours less than or greater than these risk levels may also be plotted for perspective to aid in interpreting the data.

Human health and ecological risks will be computed within an exposure unit (EU) boundary and compared to unacceptable risk limits to determine whether an unacceptable risk exists within the EU. For these SWMUs, the EU consists of an area up to 2 acres in size, which represents an area likely to be traversed by a human receptor. In some cases, the EU was made smaller than 2 acres to better represent what is believed to be a more realistic site worker scenario. If contamination extends beyond the EU boundary, the EU may be either reshaped or relocated to coincide with the contaminated area, or additional EUs may be established to encompass contamination extending beyond the chosen area. Any reshaping or relocation of the EU will not change the size of the EU materially, because the EU size is based on receptor behavior. For ground water and soils, site conditions are not expected to change during the time frame of this investigation, so no particular temporal considerations are necessary.

For surface water and sediment, the ability to collect samples will be affected by rain events. Sampling will be timed to coincide with rain events when high-flow surface water samples are collected. Base flow sampling will not be timed to coincide with any precipitation events.

All of these facets of decision making are presented in detail below, with flow charts, where appropriate.

1.4.4.1 Definition

The decision rule is a statement that integrates DQO planning process outputs into a concise summary of how data will be interpreted when decisions are made about the site being investigated. In this case, several decision rules have been developed to address the multiple project objectives. The decision rules

form a basis for establishing a sampling plan design that enables data of the correct type, quantity, and quality to be collected for attaining project objectives. RBTLs are analyte and medium specific, so the applicable analyte- and medium-specific RBTL and EDQL will be used for a given environmental medium when decisions are made.

1.4.4.2 Decision Rules for Establishing Background Concentrations

COPCs selection requires the comparison of site data to background concentrations. Therefore, it is necessary to establish background concentrations before proceeding to the COPC evaluation step.

Background concentrations are concentrations that would exist in the absence of influence from site operations. For mobile media such as ground water, surface water, and sediment, the background concentration is represented by concentrations upstream or upgradient of the site being investigated. When upgradient concentrations cannot be obtained because of flow patterns, side-gradient (cross-gradient) concentrations are the next best choice. For soils, background concentrations are the concentrations found in soils that are not influenced by site operations. For metals, the background concentrations will be represented by soil data from the NSWC Crane Basewide Background Soil Investigation (TtNUS, 2001a). The background data set corresponding to soil having physical characteristics (i.e., grain size, depositional environment and depth) that most closely represent the SWMU samples will be used. For COPC selection, organic chemicals will be assumed to have zero concentration in the natural environment.

If data from selected locations upstream or upgradient indicate that any of those media do not represent background concentrations, the Navy may consult with U.S. EPA Region 5 to agree on the most appropriate course of action. Specific background samples for each of the media are identified in Section 3.0.

1.4.4.3 Decision Rules for Selecting COPCs

Non-Detected Chemicals

As explained in Section 1.4.1.2, all reasonable efforts were made to obtain detection limits that are low enough for concentrations less than background concentrations, RBTLs and EDQLs to be measured for each analyte. Therefore, non-detected chemicals will not be classified as COPCs. However, if a chemical is non-detected at the MDL/IDL in all the samples in a particular medium, and the MDL/IDL exceeds the risk-based level, the chemical will be qualitatively discussed in the uncertainty analysis section.

Flowcharts (Figures 1-12 through 1-16) are provided to show the step-wise logic used when identifying COPCs.

- Organic target analytes in surface water and sediment:
  - An organic target analyte is classified as a COPC if the maximum detected target analyte concentration in any site surface water (sediment) sample exceeds its RBTL (human health risk) or its EDQL (ecological risk).

- Inorganic target analytes in surface water and sediment:
  - An inorganic target analyte is classified as a COPC if

    1. The Wilcoxon Rank-Sum (WRS) test at a 5% significance level indicates that the site surface water (sediment) population has a concentration exceeding the corresponding upstream surface water (sediment) population concentration. The upstream data set used for these comparisons will be surface water (sediment) samples collected upstream of the SWMU.

       AND

    2. The maximum detected target analyte concentration in any site surface water (sediment) sample exceeds its RBTL (human health risk) **or** its EDQL (ecological risk)

- Organic target analytes in surface soil:
  - An organic target analyte is classified as a COPC if the maximum detected target analyte concentration in any site surface soil sample exceeds its RBTL (human health risk) or its EDQL (ecological risk).

- Inorganic target analytes in surface soil:
  - An inorganic target analyte is classified as a COPC if

    1. The WRS test at a 5% significance level indicates that the site surface soil population has a concentration exceeding the corresponding background surface soil population concentration. **Note:** The background data set used for these comparisons will be that soil type from the NSWC Crane Basewide Background Soil Investigation that most

closely matches the site soil samples in terms of depositional environment, depth, and grain size. If multiple soil types exist at a site, the appropriate corresponding soil type from the background data set will be used in the comparison with each site soil type.

AND

2. The maximum detected target analyte concentration in any site surface soil sample exceeds its RBTL (human health risk) **or** its EDQL (ecological risk).

- Organic target analytes in subsurface soil:
  - An organic target analyte is classified as a COPC if the maximum detected target analyte concentration in any site subsurface soil sample exceeds its RBTL.

- Inorganic target analytes in subsurface soil:
  - An inorganic target analyte is classified as a COPC if

    1. The WRS test at a 5% significance level indicates that the site subsurface soil population has a concentration exceeding the corresponding background subsurface soil population concentration. **Note:** The background data set used for these comparisons will be that soil type from the NSWC Crane Basewide Background Soil Investigation that most closely matches the site soil samples in terms of depositional environment, depth, and grain size. If multiple soil types exist at a site, the appropriate corresponding soil type from the background data set will be used in the comparison with each site soil type.

    AND

    2. The maximum detected target analyte concentration in any site subsurface soil sample exceeds its RBTL (human health risk).

- All target analytes in subsurface soil:
  - No COPCs will be selected for ecological risk considerations in subsurface soil.

- Organic target analytes in ground water:
  - An organic target analyte is classified as a COPC if the maximum detected target analyte concentration in any site monitoring well sample exceeds its RBTL.

- Inorganic target analytes in ground water:
  - An inorganic target analyte is classified as a COPC if

    1. The WRS test at a 5% significance level indicates that the site ground water population has a concentration exceeding the upgradient population concentration. **Note:** Data from all site wells of a given depth (shallow or deep) will be compared to the upgradient well concentrations associated with the corresponding depth.

       AND

    2. The maximum detected target analyte concentration in any site monitoring well sample exceeds its RBTL.

- All target analytes in ground water:
  - No COPCs will be selected for ecological risk considerations in ground water.

Initially, the SWMU 19 investigation will be focused on surface soil sampling. In brief, the analytical results will be compared against screening criteria and, if the criteria are exceeded (i.e., COPCs are identified), an evaluation of the data will be undertaken to determine whether media other than surface soils are likely to be contaminated. This evaluation will involve a consultation between the Navy and EPA Region 5. If other media warrant sampling and analysis, additional sampling and data collection will be planned for all media of interest.

The strategy will be similar to the sequential strategy used for the other three SWMUs:

- Identification of COPCs
- Evaluation of the nature and extent of contamination
- Evaluation of human health and ecological risk

1.4.4.4 Decision Rules for Establishing the Nature and Extent of COPCs

The concept of nature and extent conveys the notion that a concentration gradient decreases radially from a contamination source. If multiple contamination sources exist, multiple independent or overlapping regions of contamination will exist. This is likely for heterogeneous media such as soil. The nature and extent concept implies that a region exists within which COPC concentrations may be declared to present

a potentially unacceptable risk to receptors and outside of which unacceptable risk is not expected to exist.

A receptor's behavior translates into the receptor roaming a geographical area (or volume) called the EU. The receptor risk reflects the combined effect of exposure to both contaminated and uncontaminated regions within the EU. The relative proportions of the EU that are covered by contaminated and uncontaminated regions, and the contaminant concentrations within those areas, influence the magnitude of risk incurred. Even if multiple localized regions of elevated contaminant concentrations exist within the EU, the risk to the receptor may be acceptable. Consequently, relatively non-conservative reference values may be used to establish the extent of contamination. The computation of risk levels to receptors considers both the nature and extent of contamination and each receptor's EU. EUs may differ for individual receptors, so a representative EU is used for each receptor based on receptor behavior. The extent of contamination is based on human health risk comparisons.

If appropriate, geostatistical kriging will be used to estimate contaminant concentration boundaries in ground water and soil because it takes advantage of an entire data set and the spatial relationships among individual concentration values, rather than relying on individual data points. The appropriateness of kriging will depend on the concentration distributions, density of data values, spatial correlations, and similar characteristics, which can only be determined after the data are collected. If geostatistical kriging is not appropriate, contouring will be based solely on professional judgment and the observed COPC concentrations.

Ground Water and Soil Nature and Extent of Contaminants

The decision rules for ground water and soils are presented on Figure 1-17.

Solid samples will not be collected from bedrock or from beyond the SWMU study areas without discussions between the Navy and U.S. EPA.

Surface Water and Sediment Nature and Extent of Contamination

The extent of contamination will not be determined for any drainage channel surface water in which water is not present during the sampling event. However, sediments will be sampled, if available, regardless of the availability of water in the drainage channels.

The decision rules for surface water and sediment are presented on Figure 1-18.

Failure to Establish Nature and Extent of Contamination Within Three Sampling Rounds

If the COPC extent boundary is not identified within two rounds of sampling at SWMUs 12, 13, and 16 or within three rounds of sampling at SWMU 19, the Navy will seek consultation with the U.S. EPA to discuss the need for additional sampling. The following will be considered:

- The expected contribution to risk estimates of the as-yet unbounded region of contamination
- The practicality of obtaining samples from the unbounded region
- The number of sampling locations exceeding screening criteria
- Other factors that are pertinent to the evaluation but could not be anticipated in advance

1.4.4.5 Decision Rules for Evaluations of Risk

The human health and ecological risk assessment methodologies are summarized in Sections 1.4.2 and 1.4.3, respectively, and the related appendices. The decision rules for those methodologies are presented graphically on Figures 1-19 and 1-20. Exposure Point Concentrations (EPCs) in the figures refer to those established in Section 1.4.4.

1.5 STATISTICAL METHODOLOGY

The analysis of variance (or the ANOVA technique) is the basic approach used to compare one data set to another. For soils, this involves a comparison of background soil data to site soil data; for aqueous samples, it involves a comparison of upgradient (or cross-gradient) sample results with downgradient sample results. The ANOVA technique is used to test whether there is statistically significant evidence of contamination. There are two types of ANOVA: parametric and non-parametric. The parametric ANOVA methods make two important assumptions: the data are normally (or lognormally) distributed and the group variances are homogeneous. If either of these crucial assumptions are violated, a non-parametric test can be conducted using the ranks of the observations rather than the original observations themselves. Often, an inordinate amount of time is spent checking the validity of these assumptions, and few data sets pass tests of both assumptions (Shapiro-Wilk Test of Normality and Levene's test of Homogeneity of Variance). The efficiency of non-parametric procedures compared to parametric ANOVA is approximately 95 percent, if these assumptions are met, and can be much larger than 100 percent in cases where the assumptions are not met. Therefore, site sample results will be compared to the reference (background or upgradient) results using a non-parametric ANOVA procedure.

1.5.1 Limit of Detection / Field Duplicates

In the chemical analysis of environmental samples, some analytes may be present at concentrations that are below the lowest detectable concentration for the analytical procedure. The results are generally reported as not detected (rather than zero), and the appropriate limit of detection is given. This is interpreted to mean that the actual concentration lies between zero and the reported detection limit. The amount of data that are less than the detection limit plays an important role in selecting the method of addressing the limit of detection problem. The nondetects found at this Crane site will be replaced with the MDL divided by two prior to statistical analysis. This substitution is a compromise position that allows an insertion of a single numerical value in place of a range of values between zero and the MDL so that statistics can be done on the data. If all site observations are nondetect results, no statistical analysis is warranted because the data simply reflect the achievable detection limits divided by two. Another practice that has been adopted is to use only the original sample of field duplicate results in the statistical analysis. This maintains the integrity of the data set by always using the field samples to represent the site analyte concentrations and the QC samples to evaluate reproducibility of the data.

1.5.2 NON-PARAMETRIC ANOVA: The Wilcoxon Rank-Sum Test (aka Mann-Whitney U Test)

The following equations present a step-by-step procedure for conducting the Wilcoxon Rank-Sum test.

The null hypothesis (H_0) that is tested is

H_O *The site concentration IS NOT greater than the background (upgradient) concentration.*

The alternate hypothesis (H_A) is

H_A *The site concentration IS greater than the background (upgradient) concentration.*

If H_O is rejected, then H_A is accepted. If H_0 is not rejected, the data set is consistent with the H_0 distribution.

- Step 1. Combine the background and site data and rank the ordered values from 1 to *N*. Assume there are *n* site samples and *m* background samples so that $N = m + n$.

$$W = \sum_{i=1}^{n} E_i - \frac{1}{2}n(n+1)$$

- Step 2. Compute the Wilcoxon statistic *W*:

  where E_i are the ranks of the site samples (Large values of the statistic *W* give evidence of contamination in site soils).

- Step 3. Compute an approximate *Z-score*. To find the critical value of *W*, a normal approximation to its distribution is used. The expected value and standard deviation of *W* under the null hypothesis (i.e., no contamination exists) are given by the formulas

$$E(W) = \frac{1}{2}mn; \qquad SD(W) = \sqrt{\frac{1}{12}mn(N+1)}$$

  An approximate *Z-score* for the Wilcoxon Rank-Sum test may be calculated by the following equations:

$$Z = \frac{W - E(W) - \frac{1}{2}}{SD(W)}$$

  The factor of 1/2 in the numerator serves as a continuity correction since the discrete distribution of the statistic *W* is being approximated by the continuous normal distribution. If $n,m > 10$ and ties are present, an adjustment to the approximate *Z-score* must be made:

$$Z_{RS} = \frac{W - E(W) - \frac{1}{2}}{S'D(W)}$$

$$\text{where: } SD' \quad (W) = \left(\frac{mn}{12} \left[N + 1 - \frac{\sum_{j=1}^{g} t_j (t_j^2 - 1)}{N(N-1)} \right] \right)^{\frac{1}{2}}$$

  g = the number of tied groups and t_j is the number of tied data in the jth group.

- Step 4. For a one-tailed 95% confidence level test for H_0 versus the, reject H_0 and accept H_A if $Z_{ADJUSTED} > Z_{0.95} = +1.645$.

An example use of the Wilcoxon Rank-Sum test is included in Appendix E.

TABLE 1-1

DESCRIPTION OF ORDNANCE TEST AREA TESTING LOCATIONS, BASED ON 07-11-01 TELECONFERENCE WITH SITE HISTORIAN NSWC CRANE, CRANE INDIANA PAGE 1 OF 3

	Devices Tested	Location: Description
1	**Ground Smoke and Illumination Signals**	Firing Site B: Devices are launched straight up and down, with most contamination staying close to test pad center. Smoke would go where wind goes.
2	**Hand Signals, MK80, MK110, etc.**	Firing Site B: Devices are launched up to about 800 feet in a straight up and down trajectory. More than 90% of contamination would be expected to be within 20 to 30 feet of the test pad center. All flare material is consumed but a small lead slug will generally be recovered near the test pad center.
3	**Marine Smoke and Illumination Signals, MK 66, MK 117, etc.**	Devices are fired from Firing Site A to Firing Site B. Parachute suspended flares and illuminating candles are launched up to a vertical distance of 1,000 feet. Personnel hazards have traveled as far as the road area to the north, but most contamination should be within 20 to 30 feet of the test pad center.
4	**Ground Smoke and Illumination Signals**	Firing Sites A, B, and D: Handheld personnel discharge signals were ignited. Contamination is generally expected to be found within about 5 to 6 feet of the test pad center.
5	**Marine Location Marker/Sub Signal**	There is a 20-foot-deep tank at Firing Site A from which devices are launched. Some devices burn right at the tank. Contamination should be found within 10 to 15 feet of the tank. Phosphorus-loaded devices were ignited here. At Firing Site D, the contamination should be within about 5 to 6 feet of the test area center.
6	**Smoke Producing Devices**	Firing Site C: Smoke grenades and pots are ignited here and burned in the nearby sand trap southeast of the Firing Site building. Most contamination should be found within 3 to 4 feet of test pad center.
7	**Photoflash Cartridges**	Firing Site C: Cartridges can get moisture in them and explode unexpectedly. Cartridges contain Al powder. The cartridges are launched toward the southwest from the test area center. Most contamination would lie along a center line from the test pad center to the southwest and there should be heavy contamination at the test pad extending up to about 50 to 75 feet from the launch area. The circular area represents the range of protective area established to protect personnel in the case of a detonation and fragmentation event (maximum creditable event).
8	**Fragmentation & Offensive Grenades**	Firing Site C: Grenades are launched into the sand trap from Firing Site C using a pneumatic launcher. They contained up to 1 pound of TNT with lots of fragmentation occurring. Most contamination would be within 50 feet of the sand trap.
9	**Riot Grenades**	Firing Site C: Grenades are launched from Firing Site C into the sand trap. Little fragmentation occurs. CS gas (tear gas) is produced. Most contamination is within the sand trap. CS gas residual is expected (the personnel who go into the sand trap wear PPE such as respirators).
10	**Illuminating Grenades/Flares**	Firing Site C: Grenades are launched from Firing Site C into the sand trap. Most contamination should be found within 10 feet of the test pad center. Flares ignited here would also deposit most contamination near the test pad.

TABLE 1-1

DESCRIPTION OF ORDNANCE TEST AREA TESTING LOCATIONS, BASED ON 07-11-01 TELECONFERENCE WITH SITE HISTORIAN NSWC CRANE, CRANE INDIANA PAGE 2 OF 3

	Devices Tested	Location: Description
11	**Booby Trap, Flash, Illuminating, Detonating M115A1, etc.**	Firing Site C: Most contamination should be found within 2 to 3 feet of the test pad center.
12	**Practice Bomb Signal Cartridges**	Two locations -- west side of SWMU and an alternate site. • Alternate Site - Something comparable to a 10-gauge shotgun shell was fired from the roadside test location toward end of testing location. There is a chance of detonation near the weapon firing area but that potential is low. Most contamination would consist of smoke and an aluminum cap and a paper wad that would be expelled from the firing weapon within about 20 feet of the weapon. • West Side of SWMU – similar to Alternate Site.
13	**Smoke Warhead**	Warhead Arena: Lots of smoke and fragmentation generated here. The entire Warhead Arena is bare ground devoid of vegetation to prevent fires. Again, most contamination would be found near the center of the test area.
14	**Decoy Flares/Chaff Countermeasures**	Three locations – Super MAPI Area, MAPI Area, and wedge-shaped area near Bldg 2963. • Circular Super MAPI Area - Devices are fired toward and away from the 300-foot-high towers. Devices fired toward the towers originate from the east side of the test area near the road. Most contamination should be found in this eastern location of the circular area. • Rectangular MAPI Area, east of Firing Site A – Most contamination should be found near the 100-foot-high towers. • Wedge-Shaped Area (ship off-board countermeasures) near Building 2963 – Contamination should be found all along the line of site from the launch building (2963) to the edge of the PHZ, about 1,000 feet downrange.
15	**Chaff Countermeasures**	Two locations – Teardrop-shaped area and Pentagonal area • Teardrop-Shaped Area - Devices are fired at Firing Site A. Most contamination should be within 50 feet of the 20 foot deep tank. • Pentagonal Area near Warhead Arena– Devices are fired from Building 2963 toward the Warhead Arena, but some debris has been seen beyond the Warhead Arena. The contamination is probably distributed in hot spots that are generally no more than 15 feet in diameter.

TABLE 1-1

DESCRIPTION OF ORDNANCE TEST AREA TESTING LOCATIONS, BASED ON 07-11-01 TELECONFERENCE WITH SITE HISTORIAN NSWC CRANE, CRANE INDIANA PAGE 3 OF 3

	Devices Tested	Location: Description
16	**Bullet Impact Testing**	Two areas, one about 1,300 feet northeast of Warhead Arena, the other at the Super MAPI test area: Although armor-piercing bullets are tested here, no depleted uranium or other radioactive materials are used. The circular areas represent MCE areas that are established for the rare event of an unexpected detonation at the firing location. Devices are fired on a line of sight down the center of the wedge-shaped areas toward the southwest. Contamination associated with the bullets should be within about 15 feet to the front of the bullet impact location, about 3,000 feet downrange of the firing location. Contamination from firing the devices should be found within 50 meters of the weapon.
17	**Slow Cook-Off**	About 800 feet north of Firing Site A. Used to simulate a steam leak during which temperatures gradually increase until materials burn or detonate. Jet fuel provides the heat source. Testing is conducted in an enclosure and only smoke would be emitted. Otherwise, contamination should be confined to the enclosure. A gravel pad is present beneath tanks used in the cook-off test facility at the northwest corner of the OTA. Water contained in the test tanks is drained after testing and diverted through an unlined ditch to Boggs Creek.
18	**Fast Cook-Off**	About 1,200 feet south of Super MAPI test area. Used to simulate an onboard fire. Jet fuel provides the heat source. Most contamination should be found within about 50 feet of the test pad, although some fragments have been known to travel significantly farther. A gravel pad is present beneath tanks used in the cook-off test facility at the northwestern corner of the OTA. Water contained in the test tanks is drained after testing and diverted through an unlined ditch to Boggs Creek.
19	**Hazard Classification Testing IAW**	Warhead Arena and Fast Cook-Off: Many different detonation tests are conducted here. Up to 50 pounds net explosives weight is tested at any given time, but most net explosive weights are less than 5 pounds. Most contamination should be found near the Warhead Arena, but some fragments could travel to the edge of the PHZ. There is a steel plate at the bottom of the sand pit to prevent misfired devices (e.g., grenades) from gradually migrating downward in the sand. The plate is about 3 to 4 feet deep. Craters are filled in with dirt when they are created.
20	**Rocket Range Test Area**	Rockets were fired into a sand trap. There are two areas where contamination should be expected, the sand trap and the rocket firing location. In between, there should not be much contamination, if any. This area is old and not active. The sand trap has been "stoned in" multiple times so there is probably not much sand in it. If there is sand in it, it is toward the bottom of the trap. Depth of the sand trap is unknown.
21	**Annex**	Rockeye bomb is fired from Bldg 2923. There are three areas of expected contamination: 1. Explosive slug lands at impact trap. 2. Other devices are tested by dropping into the gun tub. 3. Contamination should also be found within about 25 feet of the steel plates to the east of the crescent-shaped berm.

TABLE 1-2

SUMMARY OF PREVIOUS INVESTIGATIONS
SWMUS 12, 13, 16, AND 19
NSWC CRANE CRANE, INDIANA
PAGE 1 OF 6

SWMU/Investigation	Summary/Conclusions
SWMU 12	
Mine Fill A Various Water Quality Studies conducted in 1972, 1979 and 1980	• Surface water and effluent collected at various times around the Mine Fill A area exhibited explosives contamination. • 1972 Results: TNT was detected in Building 160 effluent. TNT and RDX were detected in Building 157ground runoff. TNT, RDX, and HMX detected in Building 152 effluent. Sampling conducted at open well W039 showed no explosives contamination. • 1979 Results: TNT not detected in sediment samples. HMX detected at 1 ppm in one sediment sample. • 1980 Results: Influent to proposed treatment facility had very high explosives content. • Outfall 301: The only discharge limits exceeded since December 1986 is total explosives concentration. • Influent to the proposed treatment plant had very high explosives content.
Mine Fill A 1985 Soil Sampling at Buildings 153, 158, and 169	• Hot spots of explosives contamination detected around Buildings 153 and 158. • Metals, methylene chloride, and PCBs were all less than 1990 TCLP action levels at Building 169.
Mine Fill A Wastewater sampling in August 1989 and May 1991	• 1989 Results: Building 151 wastewater sample contained HMX (<1.0 ppm), RDX (4.2 ppm), and TNT (124 ppm). • 1991 Results: Building 160 scrubber blowdown samples contained TNT ranging from 284 to 555 ppm.
Mine Fill A 1984 Air Emissions Study at Building 160	• Particulate emissions were in compliance with applicable Indiana air standards.
RCRA Facility Investigation Phase II Release Assessment Work Plan (Never Implemented) 1992	<u>Soil Sampling</u> • Box House, Shaker House, and Melt House and Shaker House conveyors: samples were proposed to be analyzed for metals and explosives. • Sampling was proposed for around and underneath the 5,000-gallon fuel tank and samples were proposed for metals analysis. • TNT-contaminated water tank: Subsurface sample was proposed to be analyzed for metals and explosives. • Paint Lockers (Buildings 196, 197, and 2771): Samples were proposed to be analyzed for VOCs and metals. • Metal Buildings 2714 and 2715: Samples were proposed to be analyzed for metals and explosives. • Building 160: Samples were proposed to be analyzed for metals and explosives. • Rail Cars (Storage Tanks): Samples were proposed to be analyzed for metals and explosives. • Melt Houses (Buildings 152 and 157): Samples were proposed to be analyzed for metals and explosives.

TABLE 1-2

**SUMMARY OF PREVIOUS INVESTIGATIONS
SWMUS 12, 13, 16, AND 19
NSWC CRANE CRANE, INDIANA
PAGE 2 OF 6**

SWMU/Investigation	Summary/Conclusions
	• Casing Preparation Building (Building 155): Samples were proposed to be analyzed for VOCs, metals, PCBs, and explosives. • Wastewater Treatment Plant: Samples were proposed to be analyzed for metals and explosives. • Drainageways and Settling Basin Overflow/Discharge: Samples were proposed to be analyzed for metals and explosives. Surface Water • One sample from exit of SWMU or at confluence prior to exit of SWMU was proposed to be analyzed for explosives and metals. Groundwater • Three groundwater monitoring wells were installed: Samples were proposed to be analyzed for SVOCs, metals, and explosives.
Interim Measures Report Removal and Bioremediation of Mine Fill A Material October 2000	• Actions were completed for the removal and treatment of explosives-contaminated soil near Buildings 152, 157, and 158/159 at Mine Fill A. Investigative sampling also occurred at Buildings 151 and 160. Actions were initiated for the removal and treatment of explosives-contaminated soil near Buildings 153/154. • Excavated soils were successfully treated and returned to the site for backfill or future use.
Interim Measures Report Removal and Bioremediation of Mine Fill A Material Addendum #1	• Actions were completed for the removal and treatment of explosives-contaminated soil near B153/154. • Excavated soils were successfully treated and returned to the site for backfill or future use.
SWMU 13	
Mine Fill B NAVFAC, undated	• Boiler system in Buildings 166 and 171, which used PCB oils, released PCBs into nearby soil. Remedial action was taken but PCBs remain greater than 10 ppm.
Mine Fill B 1972 Soil Sample Study	• Soil samples collected from drainage ditches near Mine Fill B and a stream water sample near well W036 exhibited TNT and RDX contamination. • Soil samples from ditches near Building 172 in 1970 showed high explosives contamination. Contaminated soil was removed; however, subsequent sampling indicated that explosive contamination still existed.
Mine Fill B 1972 Monitoring Well Analytical Data	• Data were collected from water samples of wells W030, W036, and W039 located in the vicinity of Mine Fill B. • Explosives contamination was first detected at W036 in 1972. TNT and RDX were detected in soil and water samples. • Data indicate that the explosives concentrations decrease downgradient from W036.

TABLE 1-2

**SUMMARY OF PREVIOUS INVESTIGATIONS
SWMUS 12, 13, 16, AND 19
NSWC CRANE CRANE, INDIANA
PAGE 3 OF 6**

SWMU/Investigation	Summary/Conclusions
Mine Fill B Surface water and Effluent Sampling 1980	• North Ditch sample contained no detectable explosives. Samples from the central and south-central ditches had measurable explosives in both aqueous and sediment fractions. South Ditch samples contained no detectable explosives in water. However, high levels of RDX and HMX were found in South Ditch sediment samples.
RCRA Facility Investigation Phase II Release Assessment Work Plan (Never Implemented) 1992	**Soil Sampling** • Box Houses (Buildings 2174, 2175, 2176, and 2177): Samples were proposed to be analyzed for metals and explosives. • Shaker Houses (Buildings 167, 168, 172, and 173): Samples were proposed to be analyzed for explosives and metals. • Melt House and Shaker House Conveyor Lines: Samples were proposed to be analyzed for metals and explosives. • Paint Lockers (Building 193, 194, and 195): Samples were proposed to be analyzed for VOCs, metals, and PCBs. • Metals Building (Buildings 2502 and 2503): Samples were proposed to be analyzed for explosives and metals. • Former Thermanol Boilers: Samples were proposed to be analyzed for PCBs. • Valve Box near Building 2790: Samples were proposed to be analyzed for VOCs, metals, and PCBs. • Melt Houses (Buildings 166 and 171): Samples were proposed to be analyzed for explosives and metals. • Casing Preparation Building (Building 169) Dust Dropout: Samples were proposed to be analyzed for VOCs, metals, PCBs, and explosives. • Bag House Filter Drop Out: Samples were proposed to be analyzed for metals and explosives. • Building 169 Utility Pads: Samples were proposed to be analyzed for metals, PCBs, and explosives. • Nitrate Preparation Building: Samples were proposed to be analyzed for explosives and metals. • Former Compressor Location between Buildings 2790 and 2171: Samples were proposed to be analyzed for metals and PCBs. • Hazardous Waste Storage Lockers. Building 174, Cart Shelter/Washout, drainage ways, and Settling Basins Overflow/Discharge: Samples were proposed to be analyzed for explosives and metals. **Surface Water** • Drainageways and Settling Basin Overflow/Discharge at exit of SWMU or confluence prior to exit: Samples were proposed to be analyzed for explosives, metals. **Ground Water** • Install three ground water monitoring wells: Samples were proposed to be analyzed for SVOCs, metals, and explosives.

TABLE 1-2

**SUMMARY OF PREVIOUS INVESTIGATIONS
SWMUS 12, 13, 16, AND 19
NSWC CRANE CRANE, INDIANA
PAGE 4 OF 6**

SWMU/Investigation	Summary/Conclusions
SWMU 16	
Building 146 Discharge Sampling	• 1972 discharge water samples contained detectable levels of HMX and RDX. • 1979 discharge water samples did not contain HMX.
1980 U.S. Army Water Quality Engineering Special Study	• Sediment samples collected at Building 146 drainageways contained TNT, RDX, and HMX.
Building 146 Rotary Furnace Emissions Testing (1980 and 1981)	• Results showed that the furnace could not comply with Indiana emission standards without a baghouse on line.
1982 Rotary Kiln Ash Pile Sampling	• Ash from the rotary kiln furnaces was determined to be a RCRA toxicity characteristic hazardous waste
1982 and 1985 Building 146 Surface Soil/Sediment Sampling	• The 1982 samples from the south surface water drainage ditch exceeded the EP Toxicity action levels for cadmium and lead and contained trace levels of explosives. • One 1985 sample exceeded the EP Toxicity action level for lead. • None of the 1985 samples contained detectable levels of explosives.
October to December 1989 Surface Soil Sampling at Building 146 Incinerator Bay	• Soil results showed detectable concentrations of arsenic, barium, cadmium, chromium, lead, selenium, and silver for incinerator bay and background soil samples. • Arsenic, cadmium, chromium, selenium, and silver exceeded background concentrations. • Detectable levels of PCBs were not detected at Building 146.
Building 146 Monitoring Well Data	• Four wells (WES-14-1-83 through WES-14-4-83) were installed near Building 146 in July 1963. • Soils around Building 146 appeared to contain elevated levels of lead and zinc. • Lead and zinc detected in two wells were an order of magnitude greater than concentrations in the other two wells.
RCRA Facility Investigation Phase II Release Assessment Work Plan (Never Implemented)	**Soil Sampling** • Collection of surface grab samples from within drainageways east and southwest of Building 146 were collected and were proposed to be for SVOCs, metals, explosives, and PCBs. • Above-ground fuel storage tanks: Samples were proposed to be analyzed for SVOCs, metals, explosives, and PCBs. • Within limits of former Incinerator Ash Pile samples were proposed to be analyzed for SVOCs, metals and explosives. **Surface Water** • Settling Basins northeast and northwest of Building 146: Samples were proposed to be analyzed for explosives, metals, VOCs, SVOCs, and chloride.

TABLE 1-2

**SUMMARY OF PREVIOUS INVESTIGATIONS
SWMUS 12, 13, 16, AND 19
NSWC CRANE CRANE, INDIANA
PAGE 5 OF 6**

SWMU/Investigation	Summary/Conclusions
	Sediment • Samples at surface water sampling locations were proposed to be analyzed for explosives, metals, VOCs, and SVOCs. **Ground Water** • Five existing wells were proposed for sampling and analysis for explosives, VOCs, SVOCs, metals, and chloride.
Interim Measures Cleanup 1995	• Clean-up activities included removing, sampling, and disposing sludge from sumps and ash/slag piles; confirmation sampling of remaining soils; cleaning two sumps; and backfilling and restoring excavated areas. • Soil excavations during installation of new piping exhibited low levels of PCBs.
Voluntary Interim Measures Report 1999	• Interim measures were conducted from 1996 to 1998 to clean up TCE from water prior to discharge. TCE was getting into sumps. Soil sampling was conducted near the sumps and revealed no detections of TCE. Roof drains were disconnected from the sumps and re-routed, and existing floor drains in Building 146 were abandoned. New piping was installed in 1997 connecting the east and west sumps to the sanitary sewer system.
SWMU 19	
OTA, Annex, and Rocket Range	• No analytical are available data for groundwater or soil. • Surface water monitoring data from Outfall 006 indicate that discharge limits were exceeded twice during the period October 1984 and October 1989. Discharge rates were exceeded more frequently during the period October 1979 through September 1984.
RCRA Facility Investigation Phase II Release Assessment Work Plan (Never Implemented)	**Surface Water Sampling** • Grab samples were proposed for collection from drainageways and Boggs Creek and proposed to be analyzed for SVOCs, metals, CN, chloride, explosives, and phosphorus. • One grab sample from ponded water southwest of Demolition Test Area was proposed for collection and analysis for explosives and metals. **Sediment Sampling** • Samples were proposed for collection where Boggs Creek enters and exits the SWMU. The samples were proposed to be analyzed for SVOCs, metals, CN, chloride, explosives, and phosphorus. **Soil Sampling** • Firing Site C: Surface soil grab samples were proposed to be analyzed for SVOCs, metals, CN, PCBs, explosives, and phosphorus. • Warhead Arena: One surface runoff sample was proposed to be analyzed for SVOCs, metals, CN, PCBs, explosives, and phosphorus. • Abandon Pond: One surface composite sample was proposed to be analyzed for SVOCs, metals, CN, explosives, phosphorus, and nitrates.

TABLE 1-2

**SUMMARY OF PREVIOUS INVESTIGATIONS
SWMUS 12, 13, 16, AND 19
NSWC CRANE CRANE, INDIANA
PAGE 6 OF 6**

SWMU/Investigation	Summary/Conclusions
	• Bullet Impact Test Area: Composite soil samples were proposed for collection at the north fence, the railroad tie impact area, along the southwest fenceline area of the bullet impact area, and at the perimeter of the drop tower, the hillside north of Building 2888, launch and test pads, and along the roadway. Samples were proposed to be analyzed for SVOCs, metals, CN, explosives, and phosphorus. • Firing Site A: Samples were proposed for collection at the above-ground test tanks, adjacent to the test pads, the staging area, the slow and fast cook-off areas, the Super MAPI Test Area, and small test pad east of MAPI Test Area. The samples were proposed to be analyzed for SVOCs, metals, CN, explosives, and phosphorus. • Rockeye Bomblet Test Facility, Rockeye Bomblet Air Launch Impact Trap, Blast Furnace, Demolition Test Area: Samples were proposed to be analyzed for explosives and metals. • Saltwater Wave Tank/Phosphorus Test Area: Samples were proposed to be analyzed for SVOCs, metals, explosives, and phosphorus. • Unknown Test Area: Samples were proposed to be analyzed for SVOCs, metals, and explosives. • Small Test Tower: Samples were proposed to be analyzed for SVOCs, metals, and explosives. • Steel Test Pedestal/Observation Tower: Samples were proposed to be analyzed for metals and explosives. • Converted Gun Turrets/Test Chambers/Storage Lockers: Samples were proposed to be analyzed for metals, explosives, and SVOCs. • Gun Shield Barricade: Samples were proposed to be analyzed for metals, explosives, and SVOCs. • Sand Pit/Gravel Pit: Samples were proposed to be analyzed for metals and explosives. • Down Range: Samples were proposed to be analyzed for metals and explosives. **Ground Water** • Installation of four monitoring wells, including a soil boring at the underground test tank, was proposed. Samples were proposed to be analyzed for SVOCs, metals, CN, Cl, explosives, and phosphorus. • Installation of three monitoring wells at the Rockeye Bomblet Drop Facility was proposed. Samples were proposed to be analyzed for explosives and metals.

TABLE 1-3

PROJECT TARGET PARAMETERS AND RATIONALES
MINE FILLS A (SWMU 12) AND B (SWMU 13)
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 2

Parameter	MINE FILL A SWMU 12				MINE FILL B SWMU 13				Rationale/Intended Data Use
	ENVIRONMENTAL MEDIUM				ENVIRONMENTAL MEDIUM				
	SS/SB	SD	SW	GW	SS/SB	SD	SW	GW	
LABORATORY PARAMETERS									
Metals SW-846 6020/7470A/7471A Total	Near UAP	X	X	X		X	X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Metals SW-846 6020/7470A/7471A Dissolved			X	X(2)			X	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Explosives SW-846 Method 8330	Near UAP	X	X	X		X	X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
VOCs SW-846 8260B/8015B(1)	SB only at UAP				Near TB			Near TB	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
SVOCs SW-846 8270C/SIM/8151A	SB only at UAP				Near TB	Near TB	Near TB	Near TB	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
PCBs SW-846 8082					X				To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Dioxins/Furans SW-846 8290					X				To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Total Organic Carbon	Select spls	Select spls			Select spls	Select spls			To evaluate fate and transport of contaminants and to corroborate the presence or absence of contamination. Indication of bioavailability of chemicals in sediment.
Cation Exchange Capacity	Select spls	Select spls			Select spls	Select spls			To evaluate fate and transport of contaminants. Also, indication of bioavailability of chemicals in surface soils.
Hardness (Calculation)				X				X	To evaluate water quality and to calculate water-quality standards.
Cyanide	Near UAP								To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
pH	Select spls	Select spls			Select spls	Select spls			To obtain information on mobility of chemicals (especially metals).
AVS/SEM		Select spls				Select spls			To evaluate fate and transport of contaminants and to corroborate the presence or absence of contamination. Indication of bioavailability of chemicals in sediment.
Nitrate + Nitrite as Nitrate EPA 353.2			X	X			X	X	To evaluate fate and transport of potential contaminants as well as natural attenuation.

TABLE 1-3

PROJECT TARGET PARAMETERS AND RATIONALES
MINE FILLS A (SWMU 12) AND B (SWMU 13)
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 2

Parameter	MINE FILL A SWMU 12				MINE FILL B SWMU 13				Rationale/Intended Data Use
	ENVIRONMENTAL MEDIUM				ENVIRONMENTAL MEDIUM				
	SS/SB	SD	SW	GW	SS/SB	SD	SW	GW	
Ammonia EPA 350.1			X	X			X	X	To establish absence on presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
FIELD PARAMETERS									
Grain Size	X	X			X	X			To establish which background data set is most comparable for background comparisons. To evaluate fate and transport of potential contaminants.
Depositional Environment	X	X			X	X			To identify soil sampling locations in the horizontal direction for comparison to background concentrations classified according to depositional environment.
Dissolved Oxygen			X	X			X	X	To evaluate water quality. To evaluate natural attenuation and fate and transport.
Flow Rate			X				X		To establish transient rate and absence or presence of potential contaminants for interpretation of surface water chemical concentrations. Semiquantitative parameter only.
Oxidation-Reduction Potential (ORP)			X	X			X	X	To evaluate natural attenuation and fate and transport of potential contaminants. To evaluate water quality. To establish the reducing/oxidizing tendency of the sample matrix.
pH			X	X			X	X	Measured to establish well stabilization prior to collecting ground water samples. Generally useful for data interpretation and potential future uses.
Sample Depth/location	X	X	X	X	X	X	X	X	To identify sample locations in the vertical and horizontal direction.
Specific Conductance			X	X			X	X	To establish well stabilization prior to collecting ground water samples.
Temperature			X	X			X	X	To establish well stabilization prior to collecting ground water samples. Also measured because pH and specific conductivity are temperature dependent.
Turbidity			X	X			X	X	To establish well stabilization prior to collecting ground water samples. Measured to determine if contaminants caused by suspended solids.
Water Level				X				X	To calculate potentiometric surface, groundwater velocity and hydraulic gradient.

1. VOC samples will not be taken in the top 6 inches of the surface soil samples. VOC samples will be collected in the 1 to 2 feet bgs range.
2. Ground water samples will be analyzed for dissolved metals if NTUs less than 10 cannot be achieved during sampling.

GW – ground water.
SW – surface water.
SS – surface soil.
SB – subsurface soil.
Select spls - Select samples
SD – sediment.
Spls - samples.
TB – Therminol boiler.
UAP – Unidentified asphalt pad.

TABLE 1-4

PROJECT TARGET PARAMETERS AND RATIONALES
CAST HIGH EXPLOSIVES FILL/BUILDING 146 INCINERATOR (SWMU 16)
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 3

Parameter	Environmental Medium					Rationale/Intended Data Use
	SS	SB	SD	SW	GW	
LABORATORY PARAMETERS						
Metals SW-846 6020/7470A Dissolved				X	X[2]	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Metals SW-846 6020/7470A/7471A Total	X	X	X	X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Explosives SW-846 8330	X	X	X	X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Picrates Laucks HPLC Method	X	X	X	X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
VOCs [1] SW-846 8260B/8015B	X	X			X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
SVOCs SW-846 8270C/SIM/8151A	X	X	X	X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
PCBs SW-846 8082	Select spls	Select spls				To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Dioxins/Furans SW-846 8290	Select spls	Select spls				To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Total Organic Carbon	Select spls	Select spls	Select spls			To evaluate fate and transport of contaminants and to corroborate the presence or absence of contamination. Indication of bioavailability of chemicals in sediment.
Cation Exchange Capacity	Select spls	Select spls	Select spls			To evaluate fate and transport of contaminants. Also, indication of bioavailability of chemicals in surface soils.

TABLE 1-4

PROJECT TARGET PARAMETERS AND RATIONALES
CAST HIGH EXPLOSIVES FILL/BUILDING 146 INCINERATOR (SWMU 16)
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 3

Parameter	Environmental Medium					Rationale/Intended Data Use
	SS	SB	SD	SW	GW	
Hardness (Calculation)					X	To evaluate water quality and to calculate water-quality standards.
PH	Select spls	Select spls	Select spls			To obtain information on mobility of chemicals (especially metals).
AVS/SEM			Select spls			To evaluate fate and transport of contaminants and to corroborate the presence or absence of contamination. Indication of bioavailability of chemicals in sediment.
Nitrate + Nitrite as Nitrate EPA 353.2				X	X	To establish absence or presence and extent of contamination. Evaluate risks to potential human receptors from potentially site-related contaminants.
FIELD PARAMETERS						
Grain Size	X	X	X			To establish which background data set is most comparable for background comparisons. To evaluate fate and transport of potential contaminants.
Depositional Environment	X	X	X			To identify soil sampling locations in the horizontal direction for comparison to background concentrations classified according to depositional environment.
Dissolved Oxygen				X	X	To evaluate water quality. To evaluate natural attenuation and fate and transport.
Flow Rate				X		To establish transient rate and absence or presence of potential contaminants for interpretation of surface water chemical concentrations. Semiquantitative parameter only.
Oxidation-Reduction Potential (ORP)				X	X	To evaluate natural attenuation and fate and transport of potential contaminants. To evaluate water quality.
pH				X	X	Measured to establish well stabilization prior to collecting ground water samples. Generally useful for data interpretation and potential future uses.
Sample Depth/location	X	X	X	X	X	To identify sample locations in the vertical and horizontal direction.
Specific Conductance				X	X	To establish well stabilization prior to collecting ground water samples.
Temperature				X	X	To establish well stabilization prior to collecting ground water samples. Also measured because pH and specific conductivity are temperature dependent.

TABLE 1-4

PROJECT TARGET PARAMETERS AND RATIONALES
CAST HIGH EXPLOSIVES FILL/BUILDING 146 INCINERATOR (SWMU 16)
NSWC CRANE, CRANE, INDIANA
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Parameter	Environmental Medium					Rationale/Intended Data Use
	SS	SB	SD	SW	GW	
Turbidity				X	X	To establish well stabilization prior to collecting ground water samples. Measured to determine if contaminants caused by suspended solids.
Water Level					X	To calculate potentiometric surface, ground water velocity, and hydraulic gradient.

1. VOC samples will not be taken in the top 6 inches of the surface soil samples. VOC samples will be collected in the 1 to 2 feet bgs range.
2. Ground water samples will be analyzed for dissolved metals if NTUs less than 10 cannot be achieved during sampling.

GW – ground water.
SW – surface water.
SS – surface soil.
SB – subsurface soil.
SD – sediment.
Spls –samples.

TABLE 1-5

PROJECT TARGET PARAMETERS AND RATIONALES
PYROTECHNIC TEST RANGE (SWMU 19)
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 3

Parameter	Annex, OTA, and Rocket Range					Rationale/Intended Data Use
	Environmental Medium					
	SS	SB	SD	SW	GW(2)	
LABORATORY PARAMETERS						
Metals SW-846 6020/7470/7471ATotal	X	X	X	X	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Metals SW-846 6020/7470A Dissolved				X	X(2,3)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Explosives SW-846 8330	X	X	X	X	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
VOCs (1) SW-846 8260B/8015B	Near CO	Near CO		X(2)	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
SVOCs SW-846 8270C/SIM/8151A	Near CO	Near CO		X(2)	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Dyes	Select spls					To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Perchlorate	RR only	RR only	RR only	RR only	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Cyanide	Near CO	Near CO				To establish absence or presence and extent of contamination. Evaluate risks to potential receptors (human health and ecological) from potentially site-related contaminants.
Total Organic Carbon	Select spls	Select spls	Select spls		X(2)	To evaluate fate and transport of contaminants and to corroborate the presence or absence of contamination. Indication of bioavailability of chemicals in sediment.
Cation Exchange Capacity	Select spls	Select spls	Select spls			To evaluate fate and transport of contaminants. Also, indication of bioavailability of chemicals in surface soils.
pH	Select spls	Select spls	Select spls			To obtain information on mobility of chemicals (especially metals).

TABLE 1-5

PROJECT TARGET PARAMETERS AND RATIONALES
PYROTECHNIC TEST RANGE (SWMU 19)
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 3

Parameter	Annex, OTA, and Rocket Range					Rationale/Intended Data Use
	Environmental Medium					
	SS	SB	SD	SW	GW(2)	
AVS/SEM			Select spls			To evaluate fate and transport of contaminants and to corroborate the presence or absence of contamination. Indication of bioavailability of chemicals in sediment.
Nitrate + Nitrite as Nitrate EPA 353.2				X(2)	X(2)	To establish absence or presence and extent of contamination. Evaluate risks to potential human receptors from potentially site-related contaminants.
FIELD PARAMETERS						
Grain Size	X	X				To establish which background data set is most comparable for background comparisons. To evaluate fate and transport of potential contaminants.
Depositional Environment	X	X				To identify soil sampling locations in the horizontal direction for comparison to background concentrations classified according to depositional environment.
Dissolved Oxygen				X	X(2)	To evaluate water quality. To evaluate natural attenuation and fate and transport.
Flow Rate				X		To establish transient rate and absence or presence of potential contaminants for interpretation of surface water chemical concentrations. Semiquantitative parameter only.
pH				X	X(2)	Measured to establish well stabilization prior to collecting ground water samples. Generally useful for data interpretation and potential future uses.
Sample Depth/location	X	X	X	X	X(2)	To identify sample locations in the vertical and horizontal direction.
Specific Conductance				X	X(2)	To establish well stabilization prior to collecting ground water samples.
Temperature				X	X(2)	To establish well stabilization prior to collecting ground water samples. Also measured because pH and specific conductivity are temperature dependent.
Turbidity				X	X(2)	To establish well stabilization prior to collecting ground water samples. Measured to determine if contaminants are caused by suspended solids.
Water Level				BC		To calculate potentiometric surface, groundwater velocity, and hydraulic gradient.

TABLE 1-5

PROJECT TARGET PARAMETERS AND RATIONALES
PYROTECHNIC TEST RANGE (SWMU 19)
NSWC CRANE, CRANE, INDIANA
PAGE 3 OF 3

1. VOC samples will not be taken in the top 6 inches of the surface soil samples. VOC samples will be taken from the 1 to 2 feet bgs range.
2. The need for this analysis will be determined after the data from the first round of sampling are evaluated.
3. Ground water samples will be analyzed for dissolved metals if NTUs less than 10 cannot be achieved during sampling.

BC – Boggs Creek samples only.
CO – Cook Off testing locations.
GW – ground water.
SW – surface water.
SS – surface soil.
SB – subsurface soil.
SD – sediment.
Spls – samples.
RR – Rocket Range.
OTA – Ordnance Test Area.

TABLE 1-6

AQUEOUS FIELD TARGET PARAMETERS AND METHOD DETECTION LIMITS NSWC CRANE, CRANE, INDIANA

Parameter	Detection Limit (mg/L, unless otherwise noted)
Dissolved Oxygen	0.1 mg/L
Flow Rate	NA
Oxidation Reduction Potential (ORP)	NA
pH	NA
Specific Conductance	0.02 µS/cm
Temperature	0.1°C*
Turbidity	1 NTU
Water Level	0.01 foot*

NA – Not applicable.
mg/L = milligrams per liter.
µS/cm = microSiemens per centimeter.
NTU = nephelometric turbidity unit.
°C = degrees Celsius.
*Stated value is required limit of resolution rather than a detection limit.

TABLE 1-7

DETECTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA
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PARAMETERS	CAS NUMBER	Aqueous Matrix			Solid Matrix			
		Laboratory TV [1] (ug/L)	Laboratory RL [1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory TV [1] (mg/kg)	Laboratory RL [1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)	Risk-Based Sediment Target Level [2] (mg/kg)
EXPLOSIVES								
SW-846 METHOD 8330								
1,3,5-Trinitrobenzene	99-35-4	0.35	0.65	1100	0.25	0.5	1800	1800
1,3-Dinitrobenzene	99-65-0	0.35	0.65	2.36	0.25	0.5	0.6547	0.000924
2,4,6-Trinitrotoluene (TNT)	118-96-7	0.35	0.65	2.2	0.25	0.5	16	16
2,4-Dinitrotoluene	121-14-2	0.35	0.65	1.3	0.25	0.5	0.00004	0.00004
2,6-Dinitrotoluene	606-20-2	0.35	0.65	1.3	0.25	0.5	0.00003	0.00003
2-Amino-4,6-dinitrotoluene	35572-78-2	0.35	0.65	2.2	0.25	0.5	4.7	4.7
2-Nitrotoluene	88-72-2	0.35	0.65	61	0.25	0.5	370	370
3-Nitrotoluene	99-08-1	0.35	0.65	61	0.25	0.5	370	370
4-Amino-2,6-dinitrotoluene	1946-51-0	0.35	0.65	-- [4]	0.25	0.5	4.78	4.7
4-Nitrotoluene	99-99-0	0.35	0.65	61	0.25	0.5	370	370
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	121-82-4	0.35	0.61	0.61	0.25	0.5	4.4	4.4
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	479-45-8	0.35	0.65	360	0.25	0.5	780	780
Nitrobenzene	98-95-3	0.35	0.65	3.4	0.25	0.5	0.007	0.007
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	2691-41-0	0.35	0.65	1800	0.25	0.5	3100	3100
LAUCKS HPLC METHOD								
Picric Acid	88-89-1	0.5	1	---	17	33	---	---
Picramic Acid	96-91-3	0.5	1	---	17	33	---	---
METALS								
SW-846 Method 6020								
Antimony	7440-36-0	0.1	1.0	6	0.05	0.5	0.1423	0.3
Arsenic	7440-38-2	0.2	1.0	0.045	0.1	0.5	0.39	5.9
Barium	7440-39-3	0.1	1.0	2000	0.05	0.5	1.04	82
Beryllium	7440-41-7	0.1	1.0	4	0.05	0.5	0.1	0.1
Cadmium	7440-43-9	0.7	1.0	0.66	0.35	0.5	0.00222	0.4
Chromium (total)	7440-47-3	0.1	2	11	0.05	1	0.4	2
Cobalt	7440-48-4	0.1	2	5	0.05	1	0.14033	50
Copper	7440-50-8	0.1	2.0	5	0.05	1	2.96	16
Lead	7439-92-1	0.1	1.0	1.3	0.05	0.5	0.05373	31
Nickel	7440-02-0	0.1	2	29	0.05	1	7	7
Selenium	7782-49-2	0.4	2	5	0.2	1	0.02765	0.3
Silver	7440-22-4	0.1	2	1	0.05	1	2	0.5
Thallium	7440-28-0	0.1	1.0	0.56	0.05	0.5	0.04	0.04
Tin	7440-31-5	0.1	10	73	0.05	5	7.62	47000
Vanadium	7440-62-2	0.1	2	19	0.05	1	1.59	300
Zinc	7440-66-6	0.5	10	58.9	0.25	5	6.62	120
Aluminum	7429-90-5	2.6	200	50	1.3	100	76000	76000
Calcium	7440-70-2	2.7	300	--	1.35	150	--	--
Iron	7439-89-6	3.9	20	300	1.95	10	23000	23000
Magnesium	7439-95-4	3.7	300	--	1.85	150	--	--
Manganese	7439-96-5	0.1	3	50	0.05	1.5	1800	1800
Potassium	7440-09-7	7.4	300	--	3.7	150	--	--
Sodium	7440-23-5	1.6	300	--	0.8	150	--	--
Titanium	7441-32-6	TBD	TBD	--	TBD	TBD	--	--
Strontium	7440-24-6	0.22	0.22	22000	0.02	0.1	47000	47000
Lithium	7439-93-2	11.1	11.1	730	0.02	0.1	1600	1600
SW-846 7470A/7471A								
Mercury	7439-97-6	0.1	0.2	0.0013	0.02	0.2	0.073	0.1

TABLE 1-7

DETECTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA
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PARAMETERS	CAS NUMBER	Aqueous Matrix			Solid Matrix			
		Laboratory TV [1] (ug/L)	Laboratory RL [1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory TV [1] (mg/kg)	Laboratory RL [1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)	Risk-Based Sediment Target Level [2] (mg/kg)
VOLATILE ORGANIC COMPOUNDS								
SW-846 METHOD 8260B								
1,1,1-Trichloroethane	71-55-6	0.3	1	88	0.001	0.003	0.1	0.1
1,1,1,2-Tetrachloroethane	630-20-6	0.3	0.5	0.43	0.001	0.003	0.05	0.01089
1,1,2,2-Tetrachloroethane	79-34-5	0.3	0.5	0.055	0.001	0.003	0.0002	0.0002
1,1,2-Trichloroethane	79-00-5	0.3	0.5	0.2	0.001	0.003	0.0009	0.0009
1,2,3-Trichloropropane	96-18-4	0.3	1	0.0016	0.001	0.003	0.0014	0.0014
1,1-Dichloroethane	75-34-3	0.3	1	47	0.001	0.003	1	0.000575
1,1-Dichloroethene	75-35-4	0.3	0.5	0.046	0.001	0.003	0.003	0.003
1,2-Dibromo-3-chloropropane	96-12-8	0.3	1	0.048	0.001	0.003	0.03518	0.01998
1,2-Dibromoethane	106-93-4	0.3	0.5	0.00076	0.001	0.003	0.0069	0.0069
1,2-Dichloroethane	107-06-2	0.3	0.5	0.12	0.001	0.003	0.001	0.001
1,2-Dichloropropane	78-87-5	0.3	0.5	0.16	0.001	0.003	0.001	0.001
2-Butanone	78-93-3	0.5	5	1900	0.001	0.015	10	0.13696
2-Chloro-1,3-butadiene (chloroprene)	126-99-8	0.5	10	14	0.001	0.003	3.6	3.6
2-Hexanone	591-78-6	0.5	5	1500	0.001	0.015	12.6	1.01
4-Methyl-2-pentanone	108-10-1	0.5	5	160	0.001	0.015	443	0.54437
Acetone	67-64-1	0.5	5	610	0.001	0.015	0.8	0.45337
Acrolein	107-02-8	0.5	10	0.042	0.001	0.05	0.00023	0.0000144
Acrylonitrile	107-13-1	0.5	5	0.039	0.001	0.01	0.21	0.21
Allyl chloride (3-chloro-1-propene)	107-05-1	0.5	10	1800	0.001	0.01	3000	3000
Benzene	71-43-2	0.3	0.5	0.35	0.001	0.003	0.002	0.002
Bromodichloromethane	75-27-4	0.3	0.5	0.18	0.001	0.003	0.03	0.00113
Bromoform	75-25-2	0.3	1	8.5	0.001	0.003	0.04	0.04
Bromomethane	74-83-9	0.3	1	8.7	0.001	0.003	0.01	0.01
Carbon disulfide	75-15-0	0.3	1	84.1	0.001	0.003	0.09412	0.13397
Carbon tetrachloride	56-23-5	0.3	0.3	0.17	0.001	0.003	0.003	0.003
Chlorobenzene	108-90-7	0.3	1	10	0.001	0.003	0.07	0.06194
Chloroethane	75-00-3	0.5	1	4.6	0.001	0.003	3	3
Chloroform	67-66-3	0.3	0.3	0.16	0.001	0.003	0.03	0.027
Chloromethane	74-87-3	0.3	1	1.5	0.001	0.003	1.2	0.0000785
cis-1,2-Dichloroethene	156-59-2	0.3	1	61	0.001	0.003	0.02	0.02
cis-1,3-Dichloropropene	10061-01-5	0.3	0.3	--	0.001	0.003	0.0002	0.0002
Dibromochloromethane	124-48-1	0.3	1	0.13	0.001	0.003	0.02	0.02
Dibromomethane	74-95-3	0.3	1	61	0.001	0.003	67	67
Dichlorodifluoromethane	75-71-8	0.3	1	390	0.001	0.003	39.5	0.00133
Ethylbenzene	100-41-4	0.3	1	17.2	0.001	0.003	0.7	0.0001
Ethyl methacrylate	97-63-2	0.5	10	550	0.001	0.003	30	0.000602
Methacrylonitrile	126-98-7	0.3	1	1	0.001	0.003	0.05705	0.0000297
Methylene chloride	75-09-2	0.3	1	4.3	0.001	0.003	0.001	0.001
Methyl iodide	74-88-4	0.5	10	--	0.001	0.003	--	--
Methyl methacrylate	80-62-6	0.5	10	1400	0.001	0.03	984	0.16756
Styrene	100-42-5	0.3	1	56	0.001	0.003	0.2	0.2
Tetrachloroethene	127-18-4	0.3	1	1.1	0.001	0.003	0.003	0.003
Toluene	108-88-3	0.3	1	253	0.001	0.003	0.6	0.6
trans-1,2-Dichloroethene	156-60-5	0.3	1	100	0.001	0.003	0.03	0.03
trans-1,3-Dichloropropene	10061-02-6	0.3	0.5	0.4	0.001	0.003	0.0002	0.0002
trans-1,4-Dichloro-2-butene	110-57-6	0.5	10	--	0.001	0.01	0.0079	0.0079
Trichloroethene	79-01-6	0.3	1	1.6	0.001	0.003	0.003	0.003
Trichlorofluoromethane	75-69-4	0.3	1	1300	0.001	0.003	16.4	0.00307
Vinyl acetate	108-05-4	0.5	10	248.03	0.001	0.03	2.3	0.01295

TABLE 1-7

DETECTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA
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PARAMETERS	CAS NUMBER	Aqueous Matrix			Solid Matrix			
		Laboratory TV [1] (ug/L)	Laboratory RL [1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory TV [1] (mg/kg)	Laboratory RL [1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)	Risk-Based Sediment Target Level [2] (mg/kg)
Vinyl chloride	75-01-4	0.3	0.5	0.02	0.001	0.003	0.0007	0.0007
Total Xylenes	1330-20-7	0.3	1	117	0.001	0.01	9	1.88
SW-846 Method 8015B								
1,4-Dioxane	123-91-1	50	100	6.1	0.1	0.2	2.05	0.00000543
Acetonitrile	75-05-8	20	40	79	0.04	0.08	1.37	0.13905
Isobutanol	78-83-1	20	40	1800	0.04	0.08	20.8	3.35
Propionitrile	107-12-0	20	40	6080	0.04	0.08	0.04983	0.11466
SEMIVOLATILE ORGANIC COMPOUNDS								
SW-846 METHOD 8270C								
1,2,4,5-Tetrachlorobenzene	95-94-3	1	10	11	0.067	0.67	2.02	18
1,2,4-Trichlorobenzene	120-82-1	1	5	69.2	0.067	0.333	0.3	0.3
1,2-Dichlorobenzene	95-50-1	1	5	11	0.067	0.333	0.9	0.23132
1,3-Dichlorobenzene	541-73-1	1	5	5.5	0.067	0.333	0.13	0.13
1,4-Dichlorobenzene	106-46-7	1	2	0.5	0.067	0.333	0.1	0.1
1,4-Naphthoquinone	130-15-4	1	10	0.044	0.067	0.67	--	--
1,4-Phenylenediamine	106-50-3	1	100	6900	0.067	6.7	12000	12000
1-Naphthylamine	134-32-7	1	36	0.67	0.067	3.3	--	--
2,3,4,6-Tetrachlorophenol	58-90-2	1	25	14.06	0.067	1.7	0.19878	1.51
2,4,5-Trichlorophenol	95-95-4	1	5	3600	0.067	0.333	14	0.08556
2,4,6-Trichlorophenol	88-06-2	1	3	2	0.067	0.333	0.008	0.008
2,4-Dichlorophenol	120-83-2	1	10	18	0.067	0.333	0.05	0.05
2,4-Dimethylphenol	105-67-9	1	5	100.17	0.067	0.333	0.01	0.30453
2,4-Dinitrophenol	51-28-5	1	10	4.07	0.067	0.333	0.01	0.00133
2,6-Dichlorophenol	87-65-0	1	5	--	0.067	0.67	1.17	0.00394
2-Acetylaminofluorene	53-96-3	1	10	534.97	0.067	0.67	0.59634	0.01532
2-Chloronaphthalene	91-58-7	1	2	0.396	0.067	0.67	0.01218	0.41723
2-Chlorophenol	95-57-8	1	3	8.8	0.067	0.333	0.2	0.0117
2-Methylphenol	95-48-7	1	5	1800	0.067	0.333	0.8	0.000826
2-Naphthylamine	91-59-8	1	10	--	0.067	1.7	--	--
2-Nitroaniline	88-74-4	1	2	2.1	0.067	0.333	3.5	0.000222
2-Nitrophenol	88-75-5	1	5	13.5	0.067	0.333	1.6	0.00777
2-Picoline	109-06-8	1	20	3790	0.067	1.3	9.9	0.75305
3,3'-Dichlorobenzidine	91-94-1	1	10	0.15	0.067	0.333	0.0003	0.0003
3,3'-Dimethylbenzidine	119-93-7	1	50	0.0073	0.067	3.3	0.053	0.053
3-Methylcholanthrene	56-49-5	1	10	0.0891	0.067	0.67	0.07794	8190
3-Methylphenol [5]	108-39-4	1	10	1800	0.067	0.333	3.49	0.000845
4-Methylphenol [5]	106-44-5	1	10	180	0.067	0.333	3.49	0.000808
3-Nitroaniline	99-09-2	1	5	2.1	0.067	0.333	3.5	3.5
4,6-Dinitro-2-methylphenol	534-52-1	1	10	2.3	0.067	0.333	0.14408	0.01038
4-Aminobiphenyl	92-67-1	1	10	--	0.067	0.67	--	--
4-Bromophenyl phenyl ether	101-55-3	1	2	1.5	0.067	0.333	--	1.55
4-Chloro-3-methylphenol	59-50-7	1	5	34.79	0.067	0.333	7.95	0.38818
4-Chloroaniline	106-47-8	1	5	150	0.067	0.333	0.03	0.03
4-Chlorophenyl phenyl ether	7005-72-3	1	5	--	0.067	0.333	--	--
4-Nitroaniline	100-01-6	1	10	2.1	0.067	0.333	3.5	3.5
4-Nitrophenol	100-02-7	1	10	35	0.067	0.333	5.12	0.00778
4-Nitroquinoline-1-oxide	56-57-5	1	100	--	0.067	6.7	--	--
5-Nitro-o-toluidine	99-55-8	1	16	2	0.067	1.7	15	15
7,12-Dimethylbenz(a)anthracene	57-97-6	1	10	0.548	0.067	0.67	16.3	66.4
Acetophenone	98-86-2	1	20	0.042	0.067	1.3	0.49	0.246
Aniline	62-53-3	1	5	0.44	0.067	0.333	0.05678	0.0000338
Aramite	140-57-8	1	10	2.7	0.067	0.67	19	0.00000111

TABLE 1-7

DETECTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA
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PARAMETERS	CAS NUMBER	Aqueous Matrix			Solid Matrix			
		Laboratory TV [1] (ug/L)	Laboratory RL [1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory TV [1] (mg/kg)	Laboratory RL [1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)	Risk-Based Sediment Target Level [2] (mg/kg)
Benzyl alcohol	100-51-6	1	10	281.24	0.067	0.333	65.8	0.03394
Bis(2-chloroethoxy)methane	111-91-1	1	5	6400	0.067	0.333	0.30209	0.34971
Bis(2-chloroethyl)ether	111-44-4	1	2	0.0098	0.067	0.333	0.00002	0.00002
Bis(2-chloroisopropyl)ether	108-60-1	1	3	--	0.067	1.7	--	--
Bis(2-ethylhexyl)phthalate	117-81-7	1	2	2.1	0.067	0.333	0.92594	0.182
Butyl benzyl phthalate	85-68-7	1	5	49	0.067	0.333	0.23889	4.19
Chlorobenzilate	510-15-6	1	5	0.25	0.067	0.67	1.8	0.86029
Diallate	2303-16-4	1	10	1.1	0.067	0.67	0.45214	0.00151
Di-n-butyl phthalate	84-74-2	1	2	3	0.067	0.333	0.14979	0.1105
Di-n-octyl phthalate	117-84-0	1	10	20	0.067	0.333	709	40.6
Dibenzofuran	132-64-9	1	5	20	0.067	0.333	290	1.52
Diethyl phthalate	84-66-2	1	2	3	0.067	0.333	23	0.00804
p-(Dimethylamino)azobenzene	60-11-7	1	10	--	0.067	1.7	--	--
a,a-Dimethylphenethylamine	122-09-8	1	50	36	0.067	4	61	61
Dimethyl phthalate	131-11-3	1	5	73	0.067	0.333	734	0.02495
Diphenylamine [6]	122-39-4	1	10	412.51	0.067	0.67	1.01	0.0346
Ethyl methane sulfonate	62-50-0	1	10	--	0.067	1.7	--	--
Hexachlorobenzene	118-74-1	1	2	0.00024	0.067	0.333	0.1	0.02
Hexachlorobutadiene	87-68-3	1	1	0.223	0.067	0.333	0.03976	0.1
Hexachlorocyclopentadiene	77-47-4	1	2	50	0.067	0.333	0.75537	0.90074
Hexachloroethane	67-72-1	1	2	4.8	0.067	0.333	0.02	0.02
Hexachloropropene	1888-71-7	1	20	20	0.067	1.3	--	0.0002
Isodrin	465-73-6	1	10	0.0309	0.067	0.67	0.00332	0.05516
Isophorone	78-59-1	1	5	71	0.067	0.333	0.03	0.03
Isosafrole	120-58-1	1	10	--	0.067	1.3	9.94	0.00412
Kepone	143-50-0	1	25	0.0037	0.067	1.7	0.027	0.00331
Methapyrilene	91-80-5	1	40	--	0.067	8.3	2.78	1.44E-05
Methyl methane sulfonate	66-27-3	1	10	--	0.067	1.3	0.31549	4.74E-06
n-Nitrosodi-n-butylamine	924-16-3	1	10	0.002	0.067	0.67	0.024	0.024
n-Nitrosodiethylamine	55-18-5	1	10	0.00045	0.067	0.67	0.0032	0.0032
n-Nitrosodimethylamine	62-75-9	1	10	0.0013	0.067	0.67	0.0000321	0.00000275
n-Nitrosodi-n-propylamine	621-64-7	1	2	0.0096	0.067	0.333	0.000002	0.000002
n-Nitrosomethylethylamine	62-75-9	1	10	0.0031	0.067	0.67	0.00166	0.00000485
n-Nitrosomorpholine	59-89-2	1	10	--	0.067	0.67	0.07057	3.70E-06
n-Nitrosopiperidine	100-75-4	1	10	--	0.067	0.67	0.00665	2.26E-05
n-Nitrosopyrrolidine	930-55-2	1	10	0.032	0.067	0.67	0.01258	0.000000908
Pentachlorobenzene	608-93-5	1	10	0.47	0.067	0.67	0.49695	1.26
Pentachloroethane	76-01-7	1	10	56.42	0.067	0.67	10.7	0.68918
Pentachloronitrobenzene	82-68-8	1	10	0.26	0.067	0.67	1.9	1.9
Phenacetin	62-44-2	1	10	6.3	0.067	0.67	11.7	0.00225
Phenol	108-95-2	1	5	100	0.067	0.333	5	0.02726
Pronamide	23950-58-5	1	25	160	0.067	0.67	0.0136	0.0016
Pyridine	110-86-1	1	10	36	0.0013	0.333	1.03	0.10617
Safrole	94-59-7	1	10	40	0.067	0.333	0.40398	0.16486
o-Toluidine	95-53-4	1	15	0.28	0.067	0.67	2	2
O,O,O-Triethyl phosphorothioate	102-86-3	1	10	58.25	0.067	0.67	--	--
8270C - SELECTIVE ION MONITORING								
2-Methylnaphthalene	91-57-6	0.02	0.2	6.2	0.25	0.5	0.7	0.0202
Acenaphthene	83-32-9	0.02	0.2	9.9	0.25	0.5	29	0.00671
Acenaphthylene	208-96-8	0.02	0.2	370	0.25	0.5	130	0.00587
Anthracene	120-12-7	0.02	0.2	0.029	0.25	0.5	51	0.0469
Benzo(a)anthracene	56-55-3	0.02	0.2	0.092	0.25	0.5	0.08	0.0317

TABLE 1-7

DETECTION LIMITS VERSUS RISK CRITERIA
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PARAMETERS	CAS NUMBER	Aqueous Matrix			Solid Matrix			
		Laboratory TV [1] (ug/L)	Laboratory RL [1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory TV [1] (mg/kg)	Laboratory RL [1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)	Risk-Based Sediment Target Level [2] (mg/kg)
Benzo(a)pyrene	50-32-8	0.02	0.2	0.0092	0.25	0.5	0.062	0.0319
Benzo(b)fluoranthene	205-99-2	0.02	0.2	0.092	0.25	0.5	0.2	0.2
Benzo(g,h,i)perylene	191-24-2	0.02	0.2	7.64	0.25	0.5	119	0.17
Benzo(k)fluoranthene	207-08-9	0.02	0.2	0.0056	0.25	0.5	2	0.24
Chrysene	218-01-9	0.02	0.2	0.033	0.25	0.5	4.73	0.0571
Dibenzo(a,h)anthracene	53-70-3	0.02	0.2	0.0016	0.25	0.5	0.062	0.00622
Fluoranthene	206-44-0	0.02	0.2	8.1	0.25	0.5	122	0.1113
Fluorene	86-73-7	0.02	0.2	3.9	0.25	0.5	28	0.0212
Indeno(1,2,3-cd)pyrene	193-39-5	0.02	0.2	0.022	0.25	0.5	0.62	0.2
Naphthalene	91-20-3	0.02	0.2	6.2	0.25	0.5	0.09939	0.0346
Phenanthrene	85-01-8	0.02	0.2	2.1	0.25	0.5	45.7	0.0419
Pyrene	129-00-0	0.02	0.2	0.3	0.25	0.5	78.5	0.053
SW-846 METHOD 8151A								
Hexachlorophene	70-30-4	0.01	0.02	0.228	0.25	0.5	0.19878	18
Pentachlorophenol	87-86-5	0.01	0.02	0.56	0.25	0.5	0.001	0.001
POLYCHLORINATED BIPHENYLS								
SW-846 METHOD 8082								
Aroclor-1016	12674-11-2	0.25	0.5	0.000029	0.25	0.5	0.67971	0.0341
Aroclor-1221	11104-28-2	0.25	0.5	0.000029	0.25	0.5	0.22	0.0341
Aroclor-1232	11141-16-5	0.25	0.5	0.000029	0.25	0.5	0.22	0.0341
Aroclor-1242	53469-21-9	0.25	0.5	0.000029	0.25	0.5	0.22	0.0341
Aroclor-1248	12672-29-6	0.25	0.5	0.000029	0.25	0.5	0.22	0.0341
Aroclor-1254	11097-69-1	0.25	0.5	0.000029	0.25	0.5	0.22	0.0341
Aroclor-1260	11096-82-5	0.25	0.5	0.000029	0.25	0.5	0.22	0.0341
DIOXINS/FURANS								
SW-846 METHOD 8290								
2,3,7,8-TCDD	1746-01-6	4.6E-6	1E-5	3.00E-07	0.25 [6]	0.5 [6]	2.00E-07	3.30E-06
1,2,3,7,8-PeCDD	40321-76-4	1.34E-5	5E-5	2.80E-07	0.25	0.5	2.00E-07	3.30E-06
1,2,3,6,7,8-HxCDD	57653-85-7	3.12E-5	5E-5	2.80E-07	0.25	0.5	2.00E-07	3.30E-06
1,2,3,4,7,8-HxCDD	39227-28-6	1.94E-5	5E-5	2.80E-07	0.25	0.5	2.00E-07	3.30E-06
1,2,3,7,8,9-HxCDD	19408-74-3	4.21E-5	5E-5	2.80E-07	0.25	0.5	2.00E-07	3.30E-06
1,2,3,4,6,7,8-HpCDD	35822-46-9	8.4E-6	5E-5	2.80E-07	0.25	0.5	2.00E-07	3.30E-06
OCDD	3268-87-9	3.17E-5	1E-4	2.80E-07	0.25	0.5	2.00E-07	3.30E-06
2,3,7,8-TCDF	51207-31-9	4.7E-6	1E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
1,2,3,7,8-PeCDF	57117-41-6	1.84E-5	5E-5	9.00E-06	0.25	0.5	3.90E-05	1.30E-08
2,3,4,7,8-PeCDF	57117-31-4	3.37E-5	5E-5	9.00E-06	0.25	0.5	7.80E-06	1.30E-08
1,2,3,6,7,8-HxCDF	57117-44-9	8.1E-6	5E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
1,2,3,7,8,9-HxCDF	72918-21-9	7.5E-6	5E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
1,2,3,4,7,8-HxCDF	70648-26-9	3.05E-5	5E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
2,3,4,6,7,8-HxCDF	60851-34-5	9.01E-5	5E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
1,2,3,4,6,7,8-HpCDF	67562-39-4	1.08E-5	5E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
1,2,3,4,7,8,9-HpCDF	55673-89-7	7.78E-5	5E-5	4.50E-06	0.25	0.5	3.90E-05	1.30E-08
OCDF	39001-02-00	3.88E-5	1E-4	1.29E-03	0.25	0.5	3.90E-05	1.30E-08
Total TCDD	---	NA	1E-5	---	NA	1E-6	---	---
Total PCDD	---	NA	5E-5	---	NA	5E-6	---	---
Total HxCDD	---	NA	5E-5	---	NA	5E-6	---	---
Total HpCDD	---	NA	5E-5	---	NA	5E-6	---	---
Total TCDF	---	NA	1E-5	---	NA	1E-6	---	---
Total PCDF	---	NA	5E-5	---	NA	5E-6	---	---
Total HxCDF	---	NA	5E-5	---	NA	5E-6	---	---
Total HpCDF	---	NA	5E-5	---	NA	5E-6	---	---
DYES								

TABLE 1-7

DETECTION LIMITS VERSUS RISK CRITERIA
NSWC CRANE INDIANA
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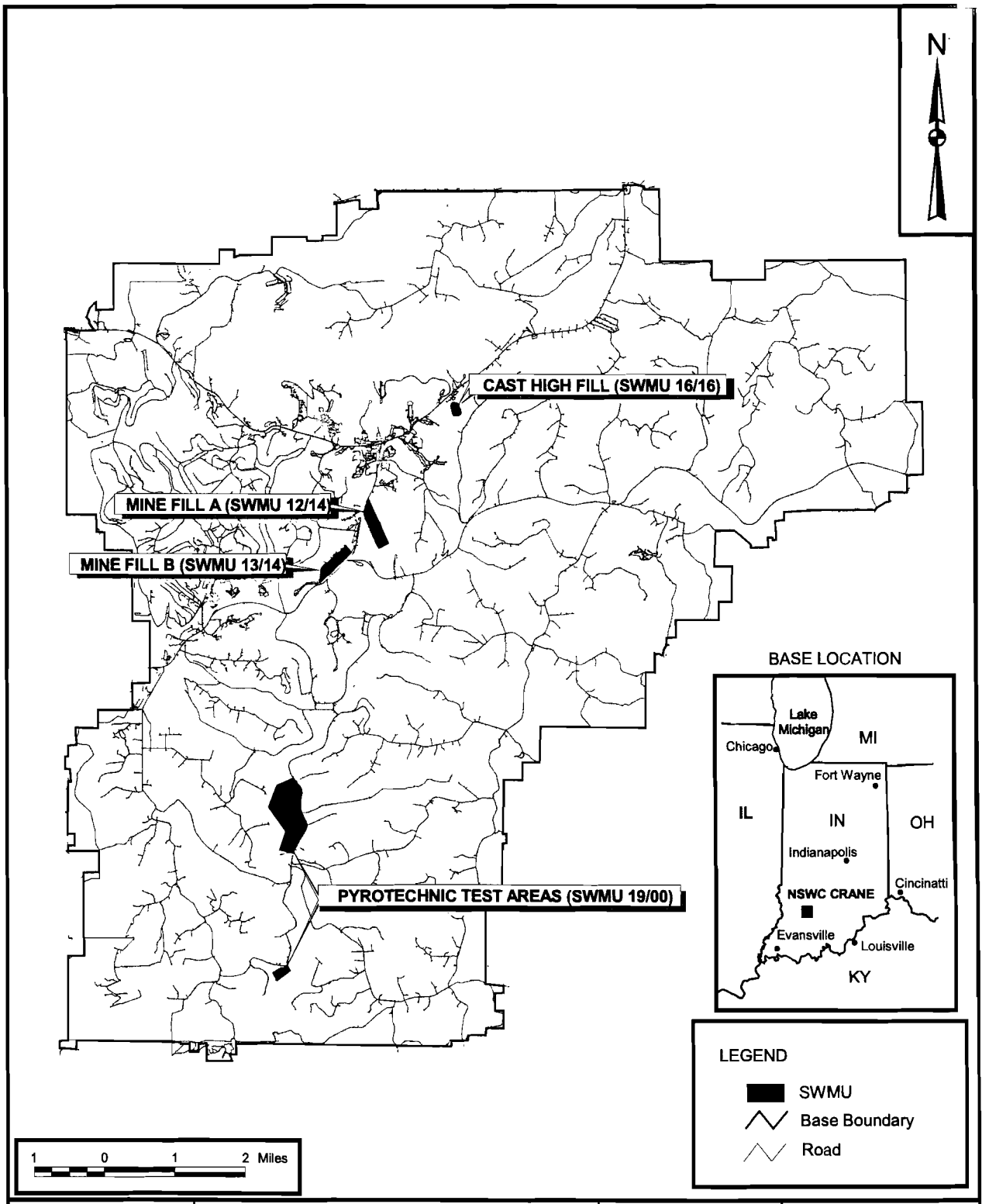
PARAMETERS	CAS NUMBER	Aqueous Matrix			Solid Matrix			
		Laboratory TV [1] (ug/L)	Laboratory RL [1] (ug/L)	Risk-Based Target Level [2] (ug/L)	Laboratory TV [1] (mg/kg)	Laboratory RL [1] (mg/kg)	Risk-Based Soil Target Level [2] (mg/kg)	Risk-Based Sediment Target Level [2] (mg/kg)
NSWC CRANE HPLC METHOD [8,9]								
Acid Blue 45		26.7	23.3	--	0.8	14.0	--	--
Acid Blue 1		43.8	26.6	450	0.6	18.0	10	--
Acid Yellow 3		24.6	22.9	--	0.1	13.7	--	--
Basic Yellow 2		170.0	23.1	60	1.4	13.9	1.3	--
Acid Yellow 23		12.8	25.1	--	0.4	15.1	--	--
Acid Yellow 73		13.5	22.6	--	0.2	13.6	--	--
Acid Orange 10		13.0	25.1	100	0.5	15.1	6.46	--
Disperse Violet 1		11.1	16.3	--	0.5	9.8	--	--
Solvent Yellow 33		13.7	17.5	--	0.1	10.5	--	--
Disperse Blue 14		16.7	18.7	--	0.9	11.2	--	--
Acid Red 64		12.1	62.5	--	0.2	37.5	--	--
Acid Blue 9		25.1	22.6	230000	0.1	13.6	1630	--
Basic Violet 10		15.0	19.9	570	0.2	11.9	12	--
2-Aminoanthraquinone		12.6	23.4	2.01	0.1	14.0	14.7	--
Solvent Orange 3		11.7	16.0	600	0.1	9.6	124	--
1-Aminoanthraquinone [10]		12.2	16.1	2.01	0.4	9.6	14.7	--
Solvent Yellow 14		13.5	17.7	460	0.1	10.6	3.23	--
Solvent Orange 7		6.1	18.0	730	0.8	10.8	52	--
Solvent Yellow 3		16.2	18.9	0.02	0.4	11.3	0.13	--
Solvent Red 1		7.9	29.0	INS	2.3	17.4	INS	--
Disperse Red 9		14.8	16.5	--	0.2	9.9	--	--
Solvent Yellow 2		11.8	20.0	0.01	0.2	12.0	0.11	--
Solvent Green 3		18.5	18.4	--	1.2	11.0	--	--
Solvent Red 24		2.9	7.1	--	0.0	4.3	--	--
MISCELLANEOUS PARAMETERS								
TOC SW-846 Method 9060/Lloyd Khan	---	TBD	TBD	---	TBD	TBD	---	---
Perchlorate EPA Method 314.0	14797-73-0	TBD	TBD	---	TBD	TBD	---	---
Hardness Standard Method 2340B	---	NA	NA	---	NA	NA	---	---
pH SW-846 Method 9045C	---	NA	NA	---	NA	NA	---	---
CEC SW-846 Method 9081	14797-65-0	NA	NA	---		0.1	---	---
Cyanide SW-846 Method 9012A	57-12-5	TBD	TBD	5.2	TBD	TBD	1.33	0.0001
Acid Volatile Sulfide/Simultaneously Extracted Metals	---	TBD	TBD	---	TBD	TBD	---	---
Nitrate + Nitrite as Nitrate EPA 353.2	14797-55-8	20	20	1000	NA	NA	---	---
Ammonia EPA 350.1	7664-41-7	50	50	---	TBD	TBD	---	---

IDL = instrument detection limit.
TV = threshold value.
µg/L = micrograms per liter.
mg/kg = milligrams per kilogram.

TBD = To Be Determined.
NA = Not Applicable
RL = Reporting Limit

1 Threshold values and reporting limits may change prior to commencement of sampling activities.
2 Value is based on the lowest human health or ecological risk-based criteria as presented in Appendix B, Table B-1 (aqueous) and B-2 (solids).
3 Shading indicates those chemicals for which the threshold value and/or reporting limit exceeds the risk based target level for the project.
4 Risk-based target level is not provided because human health or ecological risk-based criteria are not available for this compound.
5 3-Methyl and 4-methylphenol co-elute; therefore, one analytical result for 3-, 4-methylphenol will be reported.
6 N-Nitrosodiphenylamine is more toxic than diphenylamine. However, n-nitrosodiphenylamine rapidly degrades to diphenylamine. Therefore, only diphenylamine will be reported, but results for diphenylamine will be treated as n-nitrosodiphenylamine during risk assessment.
7 Units for dioxins/furans are in µg/Kg.
8 Aqueous values are based on the long gradient low-level method. Soil values are based on the short gradient method.
9 Risk values were developed by U.S. EPA Region 5 specifically for CTO 10, SWMU 2, Dye Burial Ground.
10 These risk values were developed using 2-aminoanthraquinone as a surrogate.



DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NUMBER	OWNER NO.
J. LAMEY	8/27/01		—	—
CHECKED BY	DATE	SITE LOCATION MAP SWMUs 12/14, 13/14, 16/16, AND 19/00 NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY	DATE
—	—		—	—
COST/SCHEDULE-AREA			APPROVED BY	DATE
			—	—
SCALE			DRAWING NO.	REV
AS NOTED			FIGURE 1-1	0

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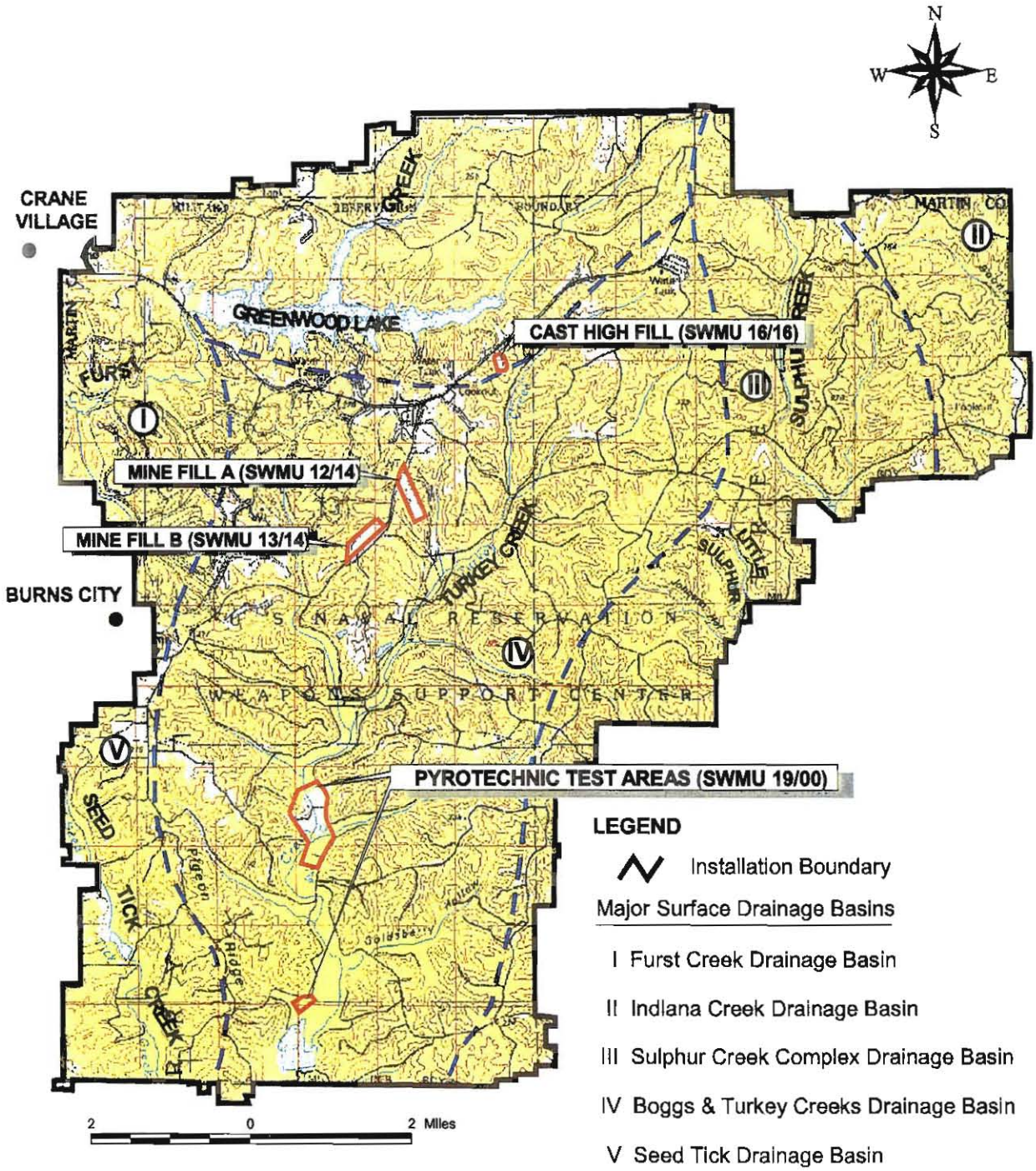

SOURCE: "Initial Assessment of Study of Naval Weapons Support Center Crane, Indiana." Naval Energy and Environmental Support Activity, May 1983.

DRAWN BY J. LAMEY — DATE 8/27/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER —	OWNER NO. —
CHECKED BY — DATE —	SWMU LOCATIONS AND DRAINAGE BASINS NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY — DATE —	
COST/SCHEDULE-AREA		APPROVED BY — DATE —	
SCALE AS NOTED		DRAWING NO. FIGURE 1-2	REV 0

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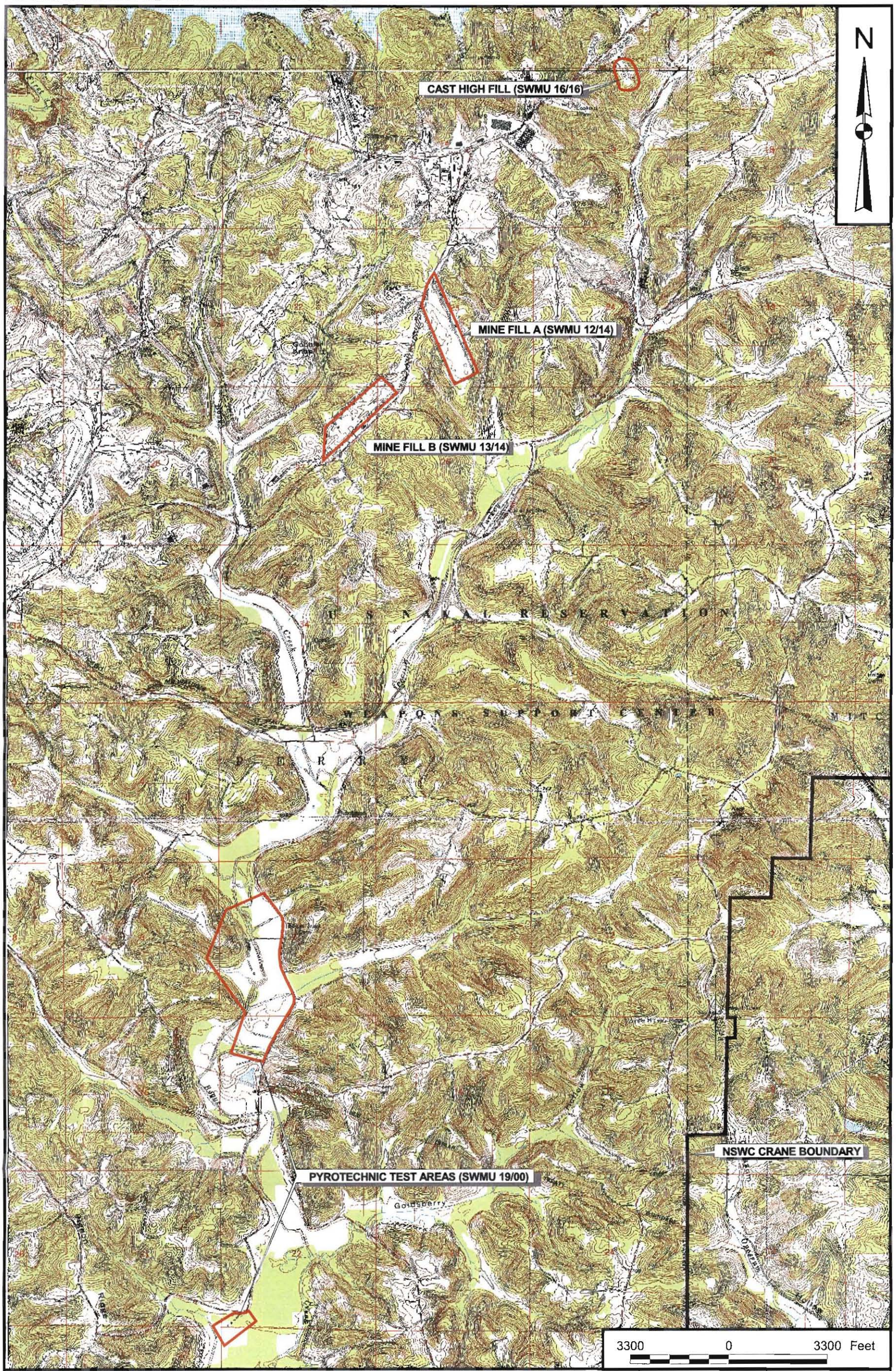
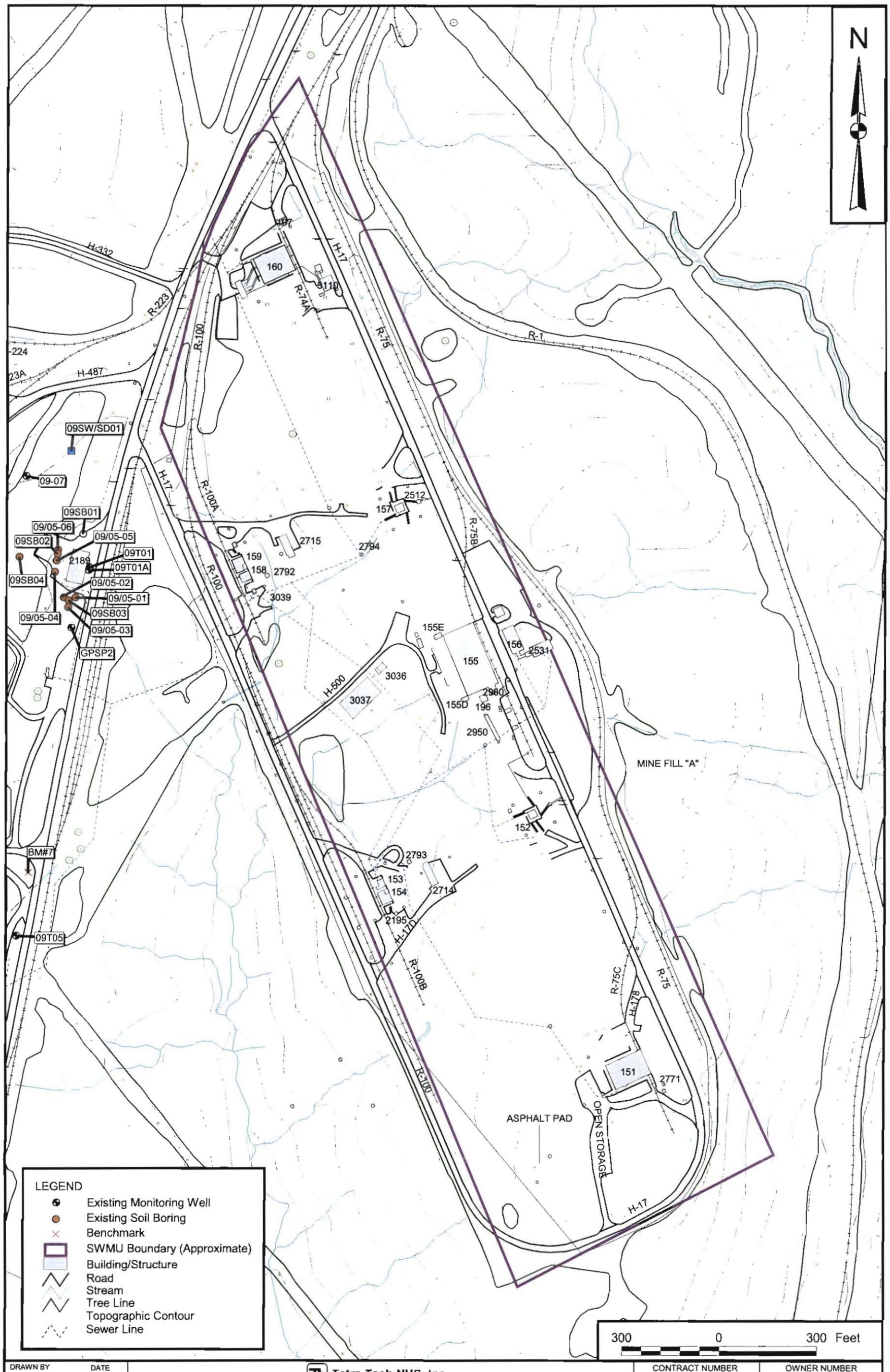
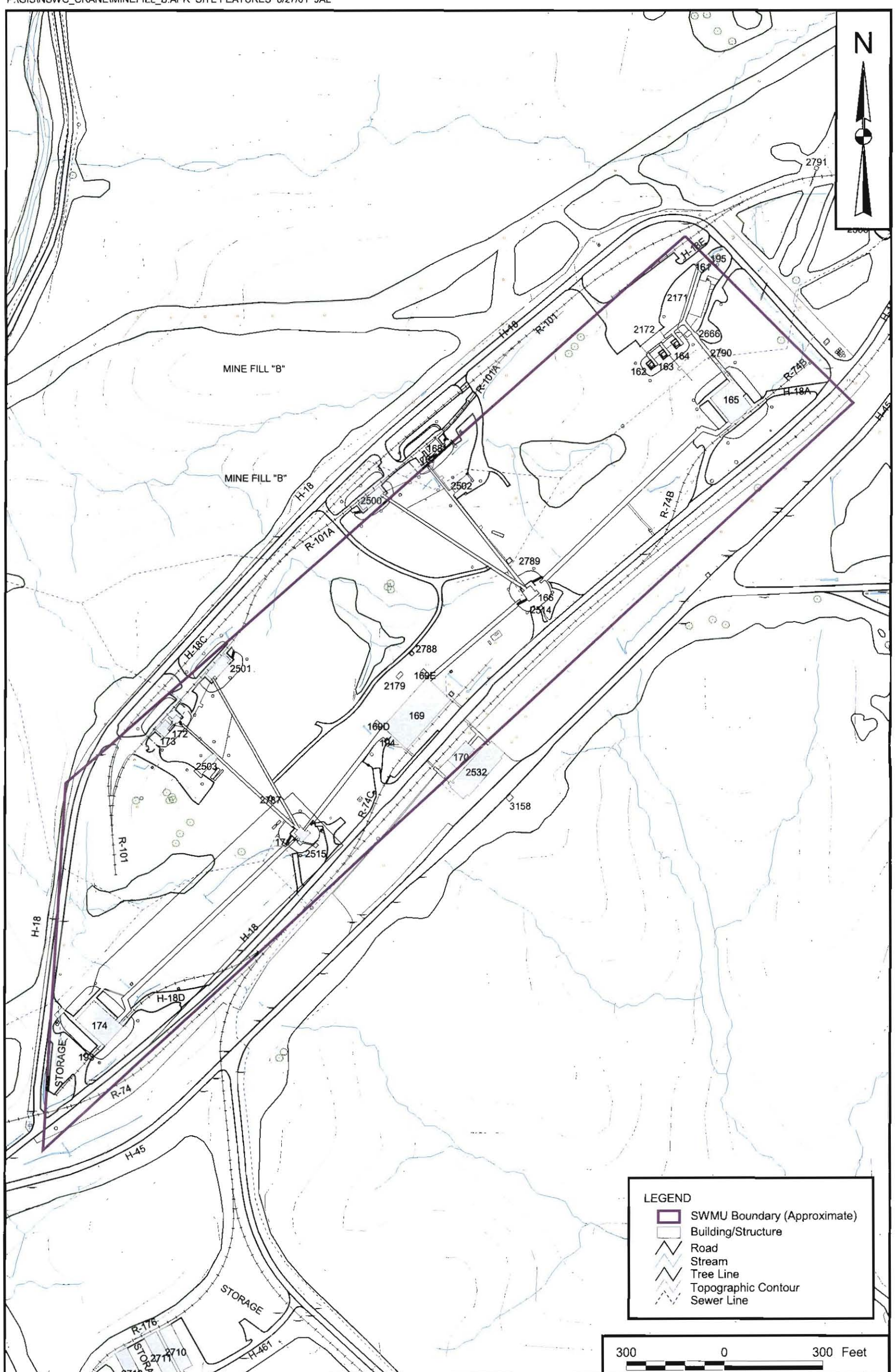


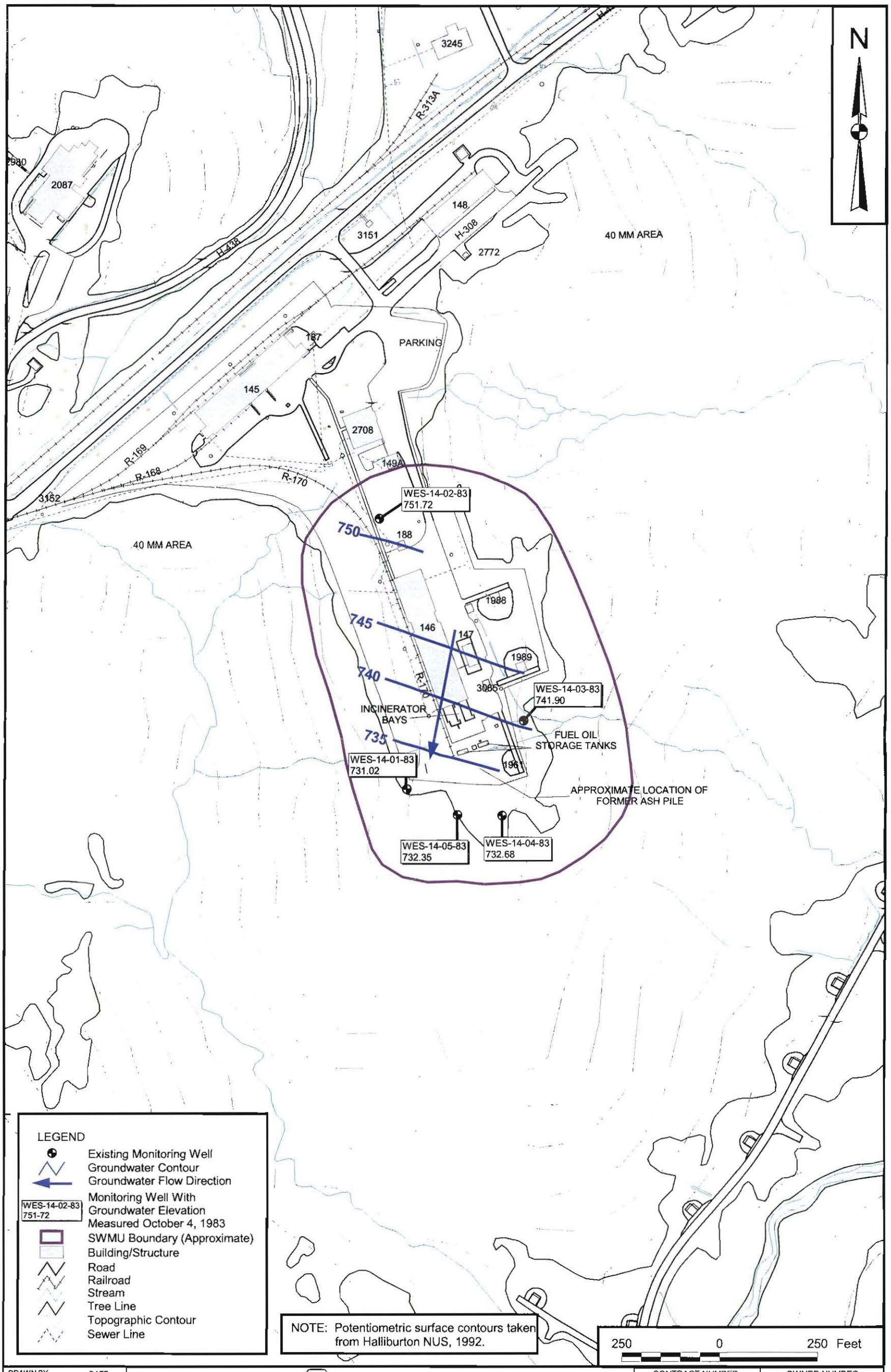
DRAWN BY J. LAMEY	DATE 8/27/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER — / OWNER NUMBER —
CHECKED BY —	DATE —	SITE MAP SWMUs 12/14, 13/14, 16/16, AND 19/00 NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY — / DATE —
COST/SCHEDULE-AREA			APPROVED BY — / DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-3 / REV 0



DRAWN BY J. LAMEY	DATE 8/20/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER —	OWNER NUMBER —
CHECKED BY	DATE	SITE FEATURES MINE FILL A (SWMU 12/14) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY —	DATE —
COST/SCHEDULE-AREA			APPROVED BY —	DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-4	REV 0

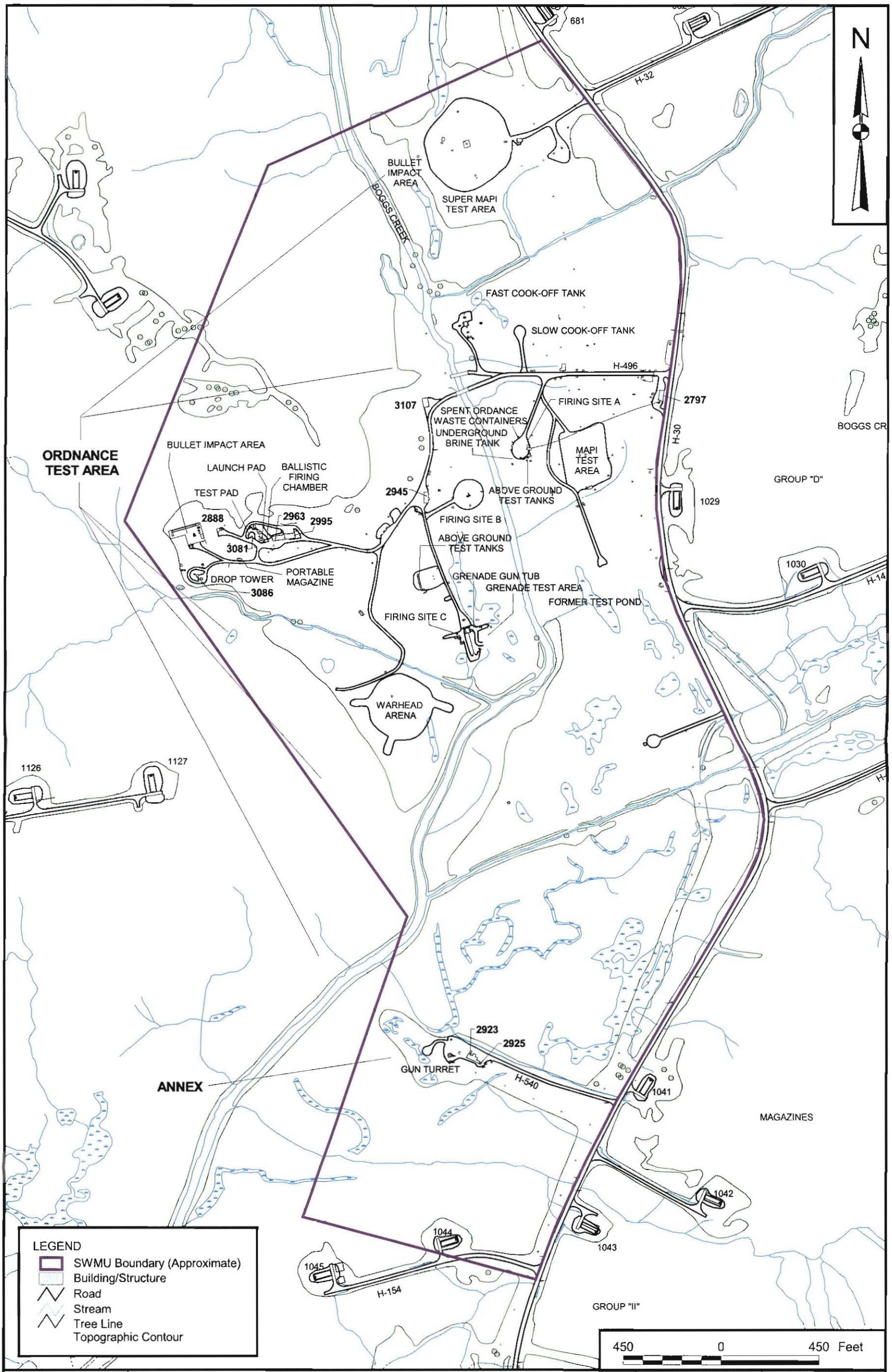


DRAWN BY J. LAMEY	DATE 8/21/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER — / OWNER NUMBER —
CHECKED BY	DATE	SITE FEATURES MINE FILL B (SWMU 13/14) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY — / DATE —
COST/SCHEDULE-AREA			APPROVED BY — / DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-5 / REV 0



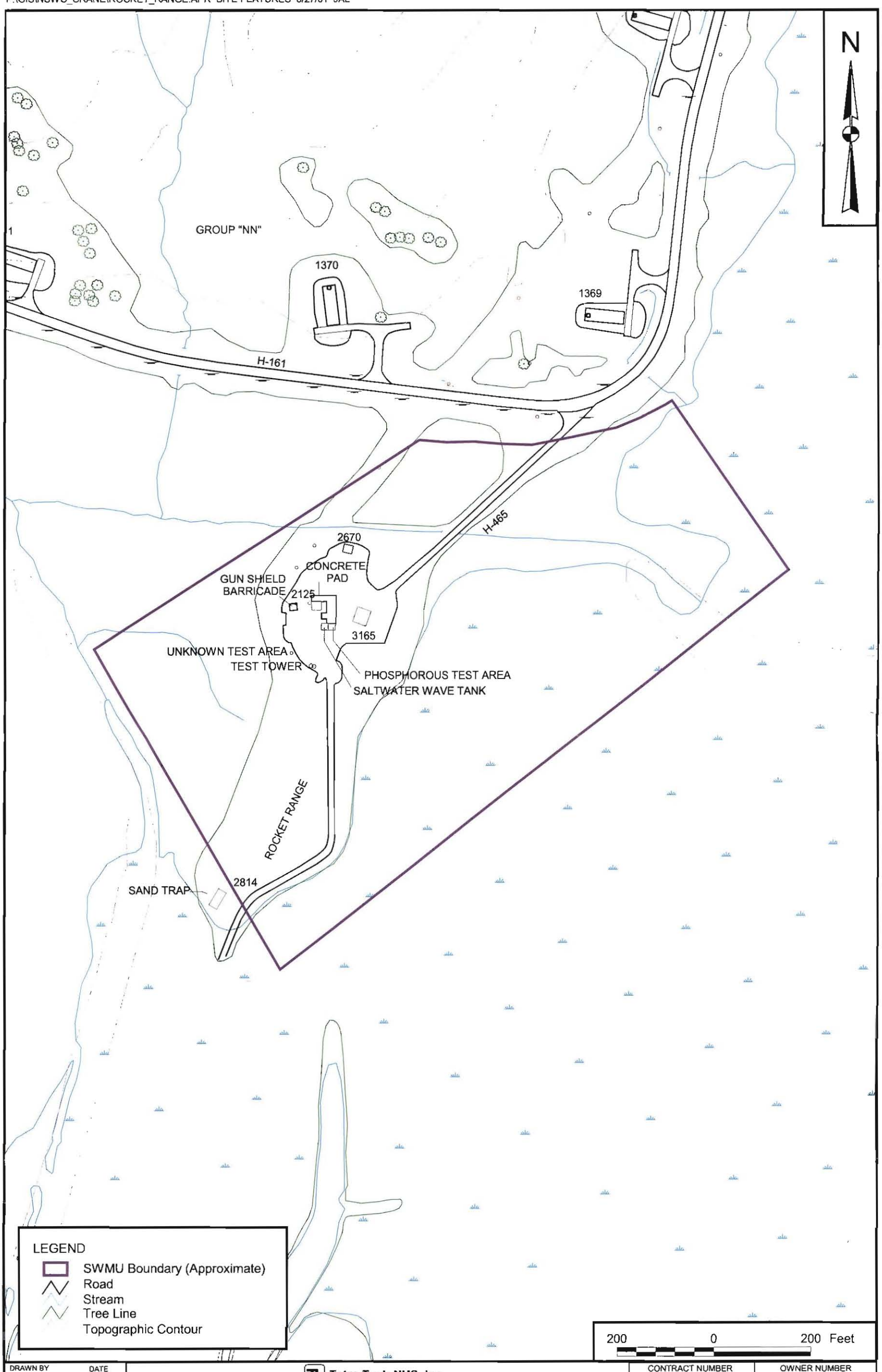
DRAWN BY J. LAMEY	DATE 8/22/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER —	OWNER NUMBER —
CHECKED BY	DATE	SITE FEATURES CAST EXPLOSIVES HIGH (SWMU 16/16) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY —	DATE —
COST/SCHEDULE-AREA			APPROVED BY —	DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-6	REV 0

P:\GIS\NSWC_CRANE\PYROTECHNIC_TEST_AREA.APR SITE FEATURES 1/11/02 JAL

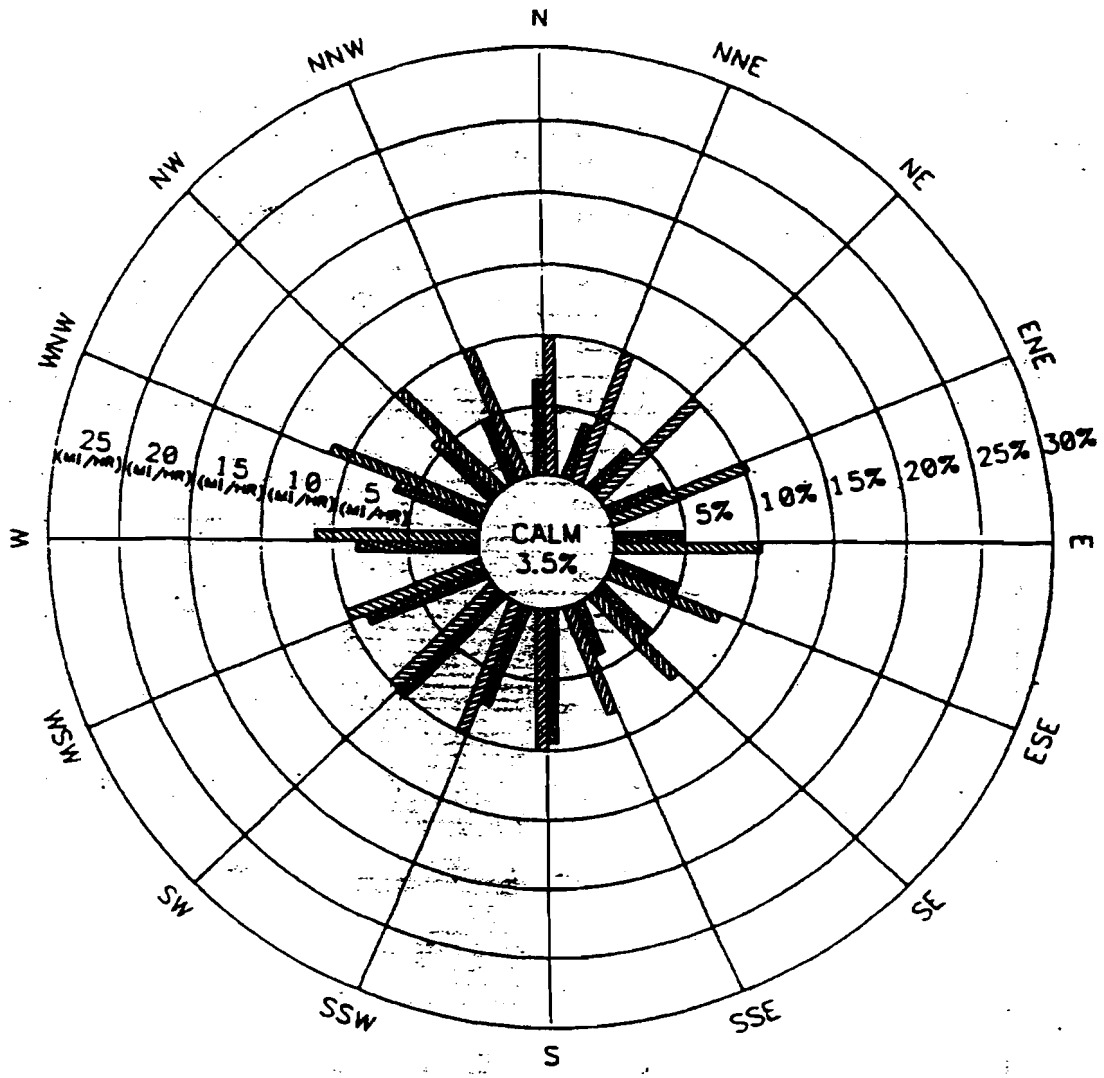


DRAWN BY J. LAMEY	DATE 8/22/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER —	OWNER NUMBER —
CHECKED BY —	DATE —	SITE FEATURES PYROTECHNIC TEST AREAS (SWMU 19/00) ORDNANCE TEST AREA AND ANNEX NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY —	DATE —
COST/SCHEDULE-AREA			APPROVED BY —	DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-7	REV 0

P:\GIS\NSWC_CRANE\ROCKET_RANGE.APR SITE FEATURES 8/27/01 JAL



DRAWN BY J. LAMEY	DATE 8/17/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER — / OWNER NUMBER —
CHECKED BY	DATE	SITE FEATURES PYROTECHNIC TEST AREAS (SWMU 19/00) ROCKET RANGE NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY — / DATE —
COST/SCHEDULE-AREA			APPROVED BY — / DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-8 / REV 0



LEGEND

WIND DIRECTION FREQUENCY (PERCENT)
(DIRECTION FROM WHICH THE WIND IS BLOWING)

MEAN WIND SPEED (MI/HR)

DATA SOURCE: INDIANAPOLIS, INDIANA AIRPORT

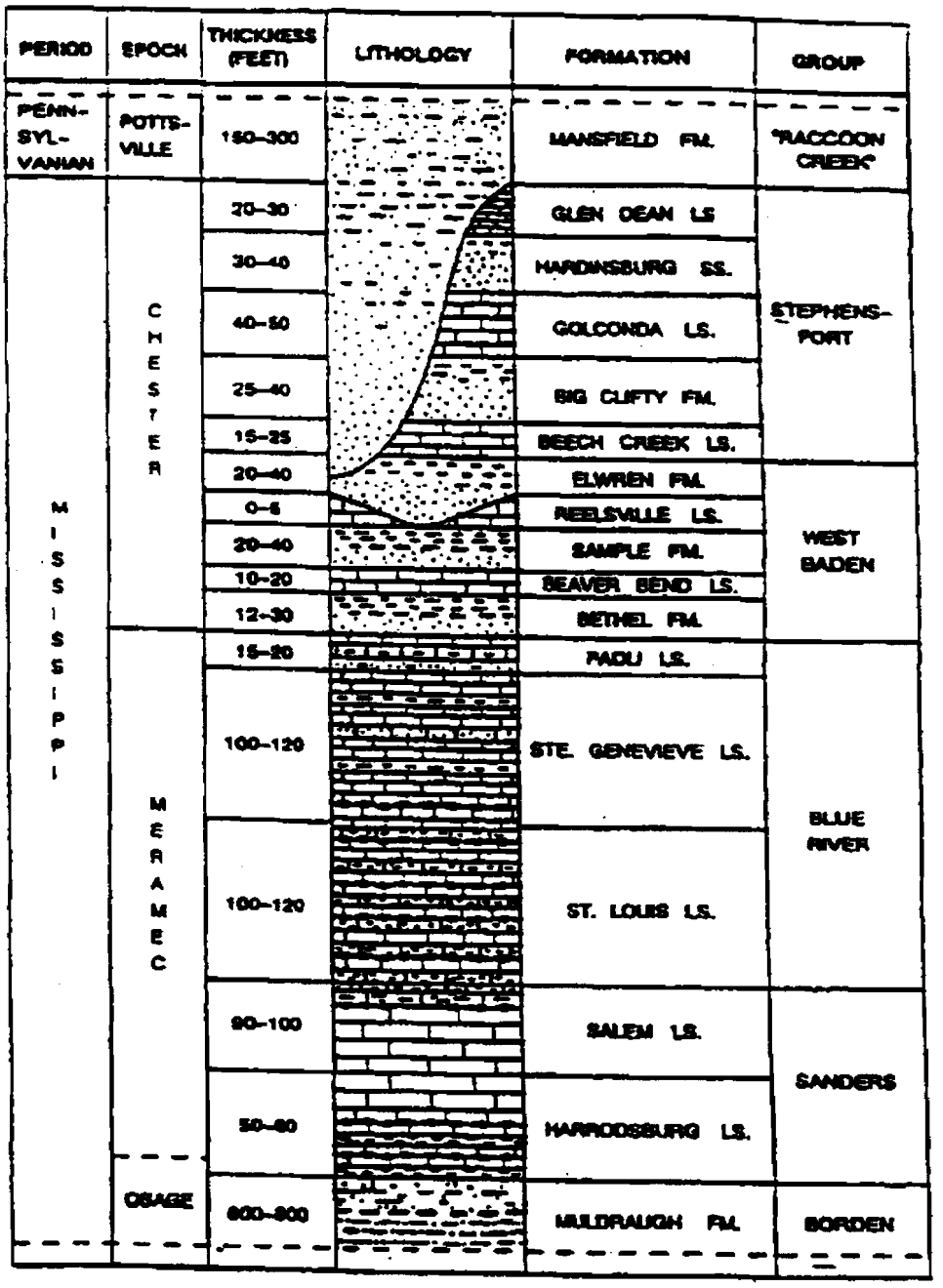
PERIOD OF RECORD: 1985-1989
43,824 OBSERVATIONS

DRAWN BY DLT DATE 6/7/00			CONTRACT NO. 0514
CHECKED BY DATE		APPROVED BY DATE	
COST/SCHED-AREA		APPROVED BY DATE	
SCALE NOT TO SCALE		DRAWING NO FIGURE 1-9	REV. 1

FIVE-YEAR WINDROSE
SUMMARY FOR INDIANAPOLIS, INDIANA
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

FORM CADD NO. 3DIV_AV.DWG - REV 0 - 1/20/98

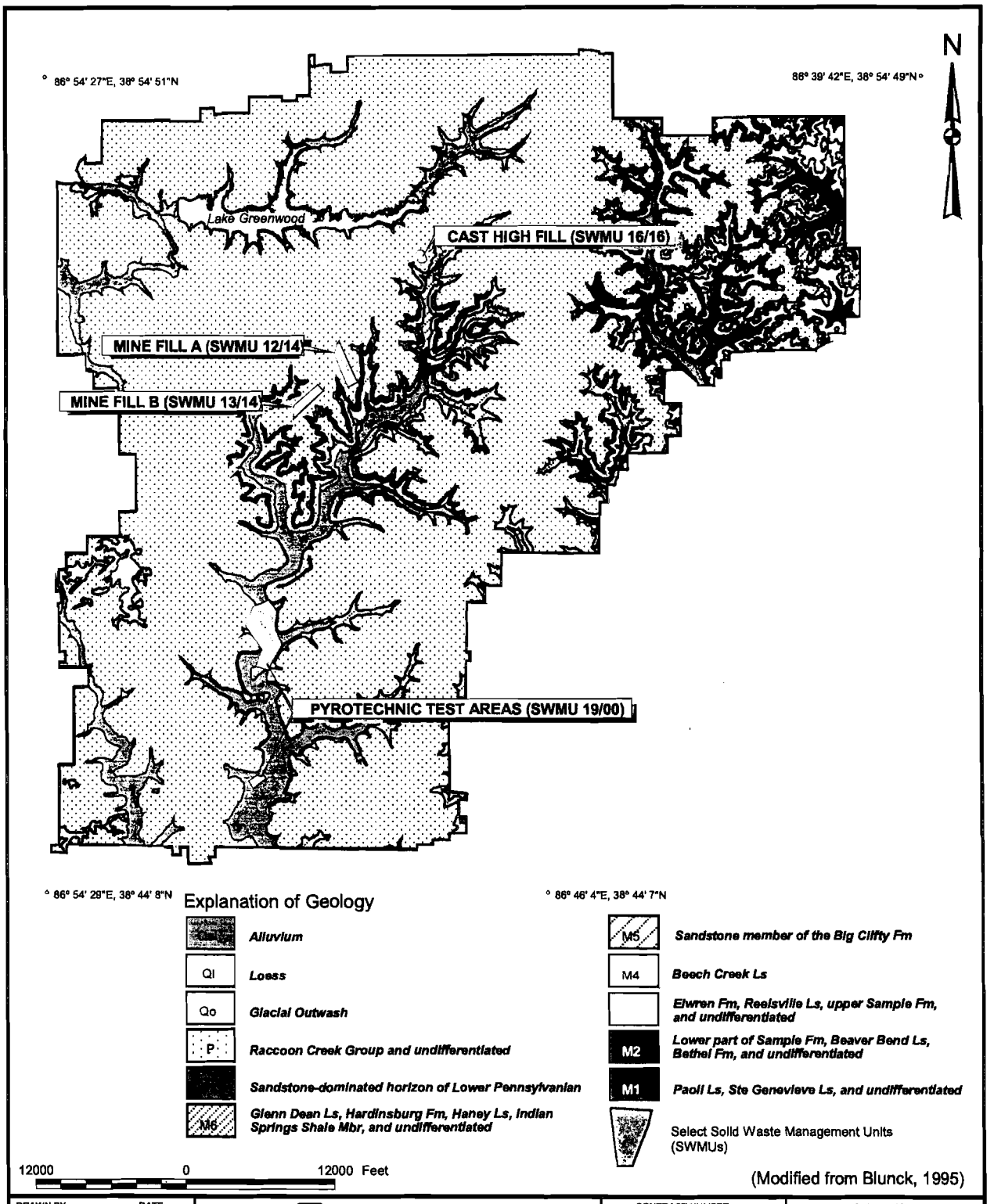
ACAD: 0514KMB1.dwg 06/07/00 D1



Source: Draft Report, RCRA Facility Investigation
Phase II Groundwater Release Assessment, SWMU 06/09
Demolition Area and Phase III Release
Characterization SWMU 07/09 Old Rifle Range
November 1995 - Figure 13
by William L. Murphy and Roy Wade

DRAWN BY J. LAMEY	DATE 8/28/01	Tetra Tech NUS, Inc.	CONTRACT NUMBER —	OWNER NO. —
CHECKED BY —	DATE —	STRATIGRAPHIC COLUMN FOR ROCK UNITS ENCOUNTERED AT THE NAVAL SURFACE WARFARE CENTER (SOURCE: PALMER, 1969; CITED IN MURPHY AND WADE, 1995) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY —	DATE —
COST/SCHEDULE-AREA			APPROVED BY —	DATE —
SCALE AS NOTED			DRAWING NO. FIGURE 1-10	REV 0

P:\GIS\NSWC_CRANE\MINEFILL_A.APR STRATIGRAPHIC COLUMN 8/28/01 JAL

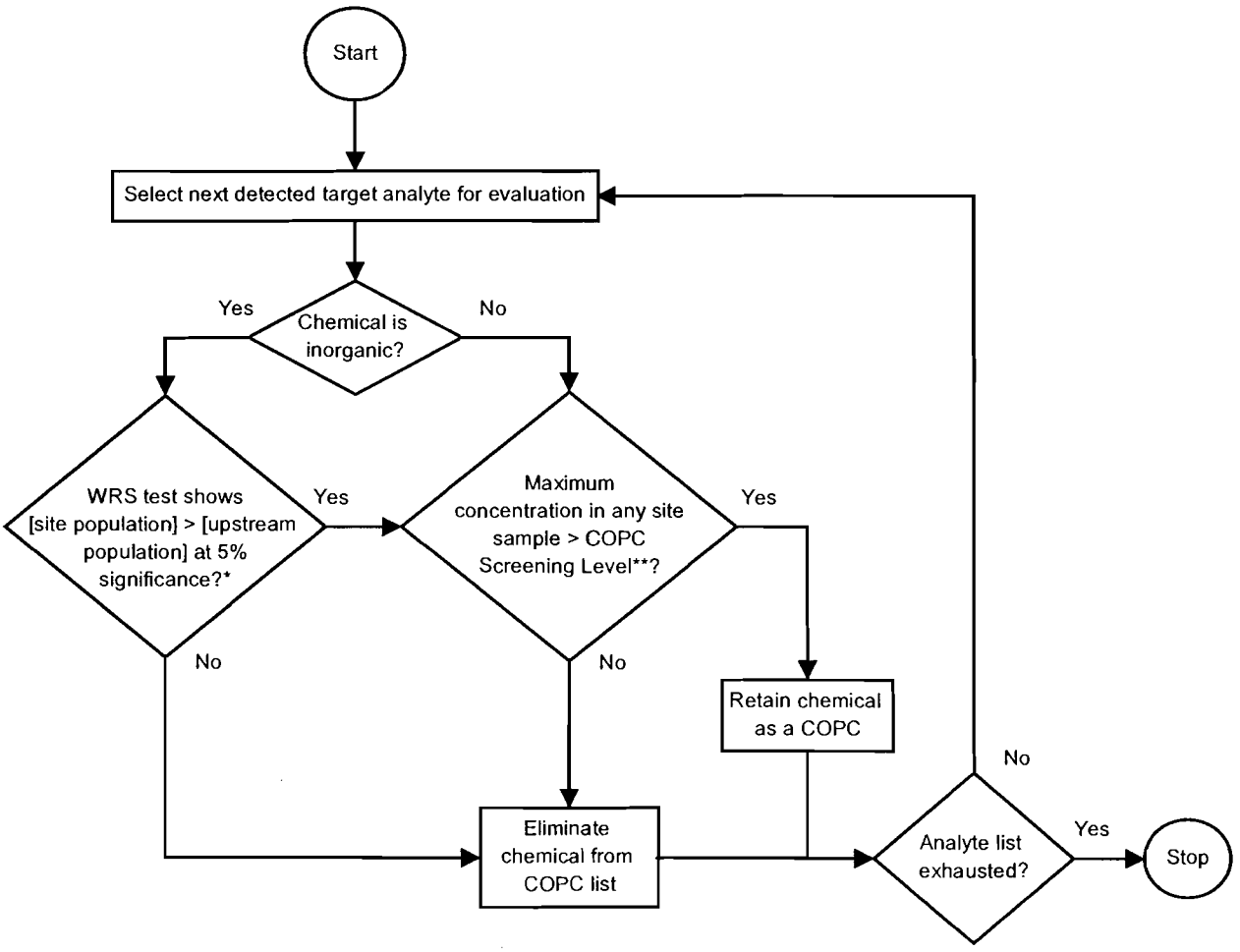


DRAWN BY	DATE	Tetra Tech NUS, Inc.	CONTRACT NUMBER	OWNER NO.
J. LAMEY	8/27/01		—	—
CHECKED BY	DATE	SURFICIAL GEOLOGY MAP NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY	DATE
—	—		—	—
COST/SCHEDULE-AREA			APPROVED BY	DATE
			—	—
SCALE			DRAWING NO.	REV
AS NOTED			FIGURE 1-11	0

P:\GIS\NSWC_CRANE\MINEFILL_A.APR SURFICIAL GEOLOGY MAP LAYOUT 8/30/01 JAL

FIGURE 1-12

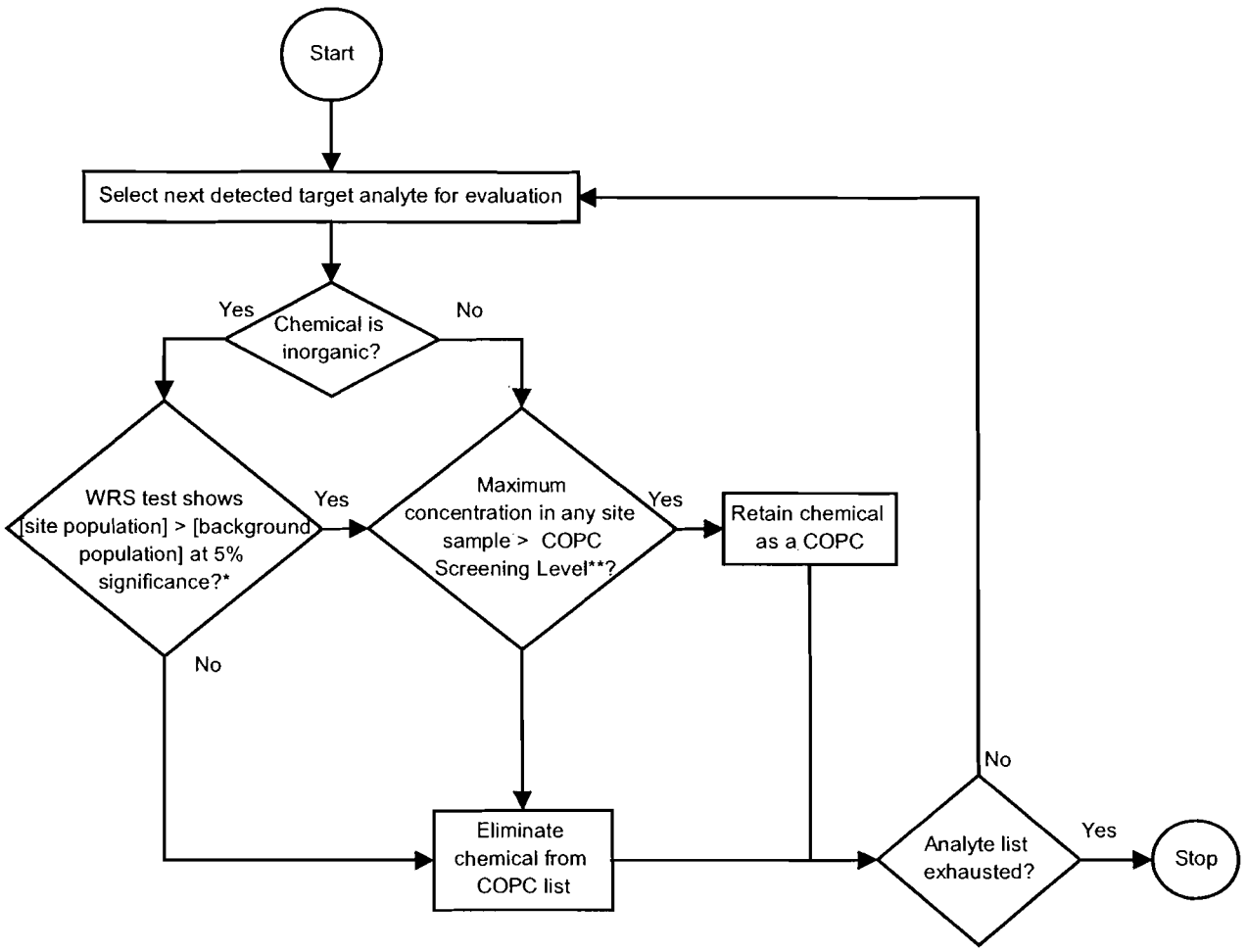
HUMAN HEALTH RISK ASSESSMENT COPC SELECTION FOR SURFACE WATER AND SEDIMENT NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
RBTL = Risk-based target level
WRS = Wilcoxon Rank Sum
*The upstream population will be represented by samples collected upstream of the SWMU investigative samples.
**See Appendix C, Section C.1.2.1, for details

FIGURE 1-13

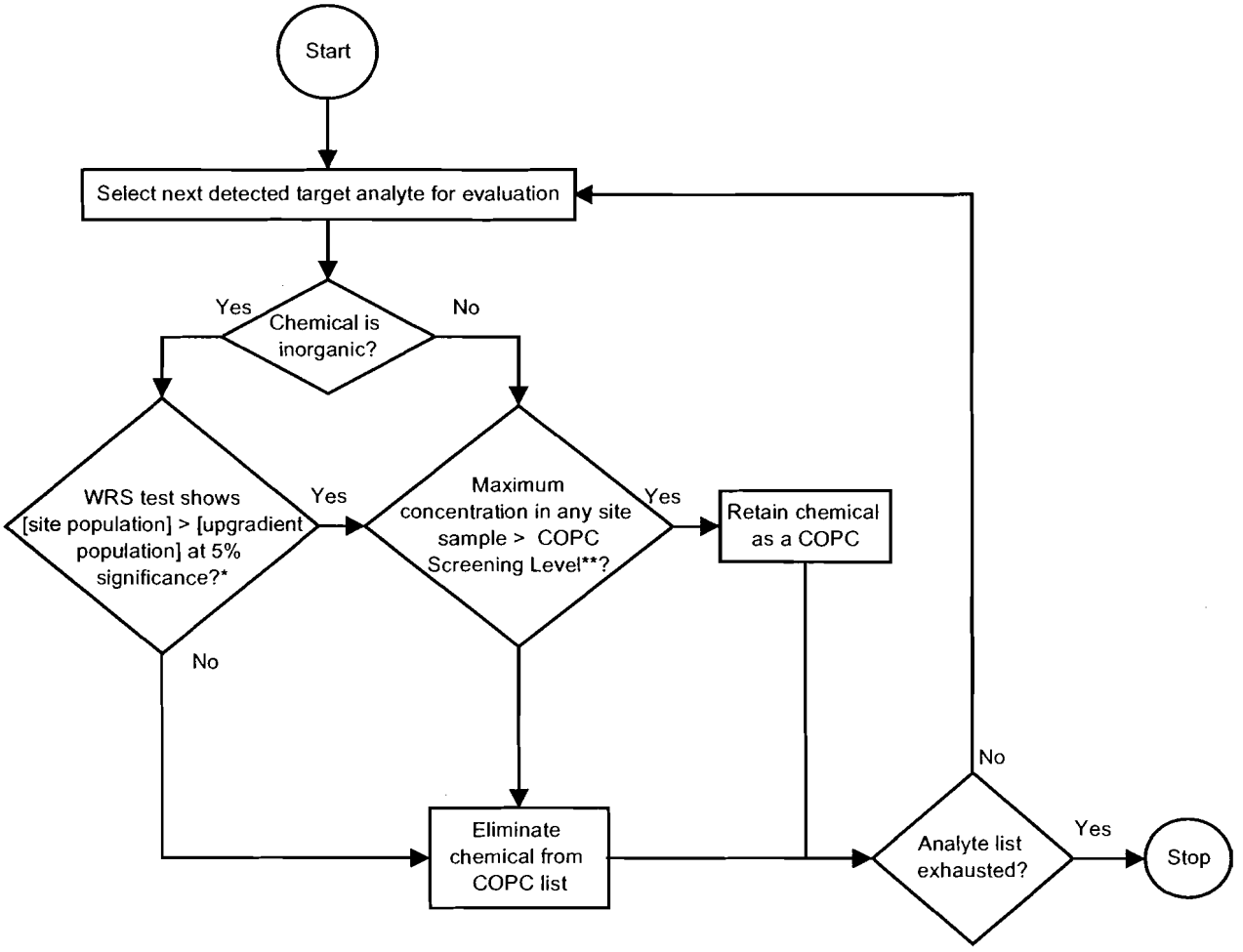
HUMAN HEALTH RISK ASSESSMENT COPC SELECTION FOR SURFACE AND SUBSURFACE SOIL NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
RBTL = Risk-based target level
WRS = Wilcoxon Rank Sum
*The background population will be represented by soil data from the NSWC Basewide Background Soil Investigation that most closely matches the site soil in terms of depositional environment, depth, and grain size. If multiple soil types are present at the site, multiple background soil types will be used, as necessary, to obtain a reasonable match for each site soil type.
**See Appendix C, Section C.1.2.1, for details.

FIGURE 1-14

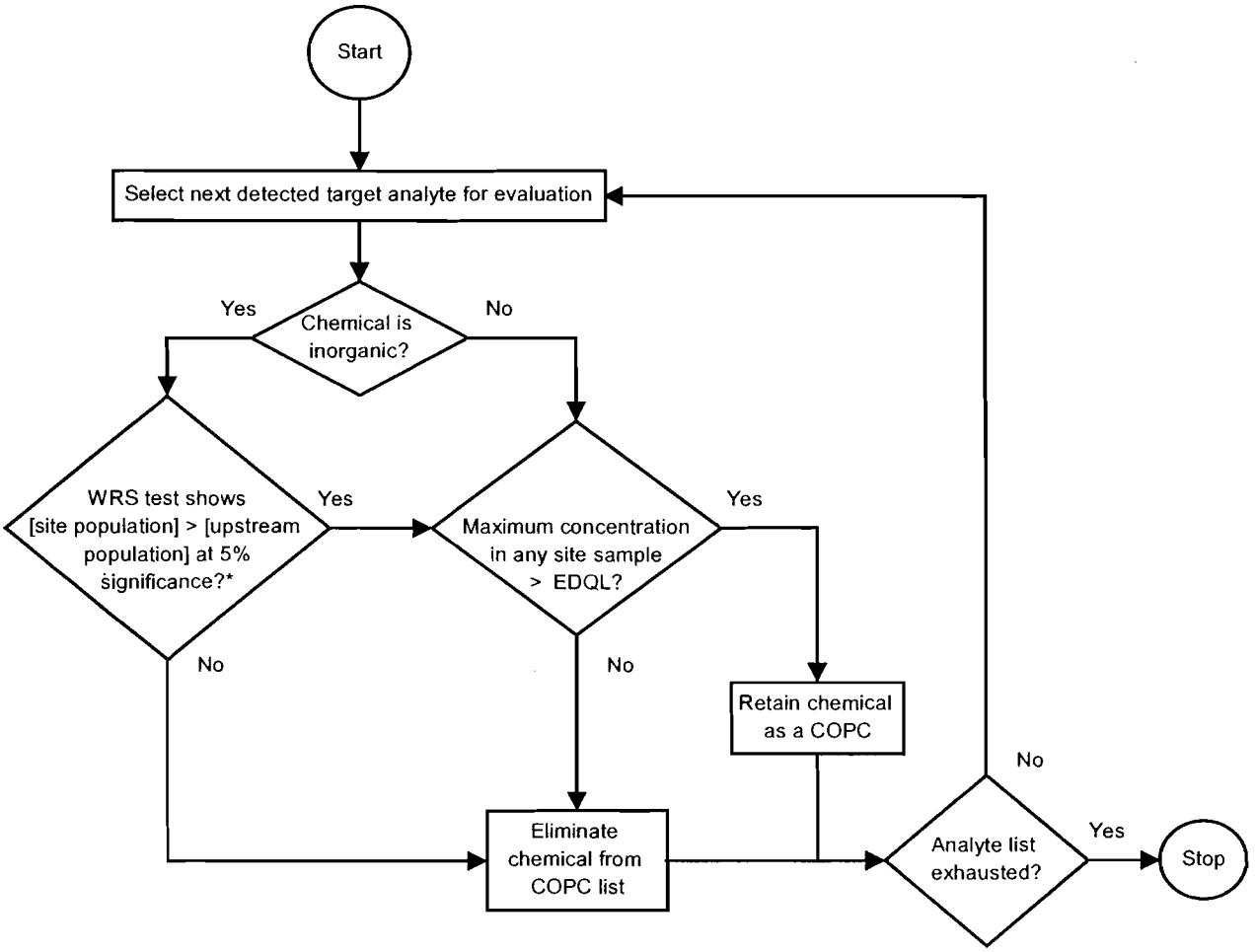
HUMAN HEALTH RISK ASSESSMENT COPC SELECTION FOR GROUND WATER NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
RBTL = Risk-based target level
WRS = Wilcoxon Rank Sum
*The upgradient population will be represented by samples from upgradient monitoring wells. Sample depths (shallow or deep) for site and upgradient will be matched to represent corresponding aquifers/depths.
**See Appendix C, Section C.1.2.1, for details.

FIGURE 1-15

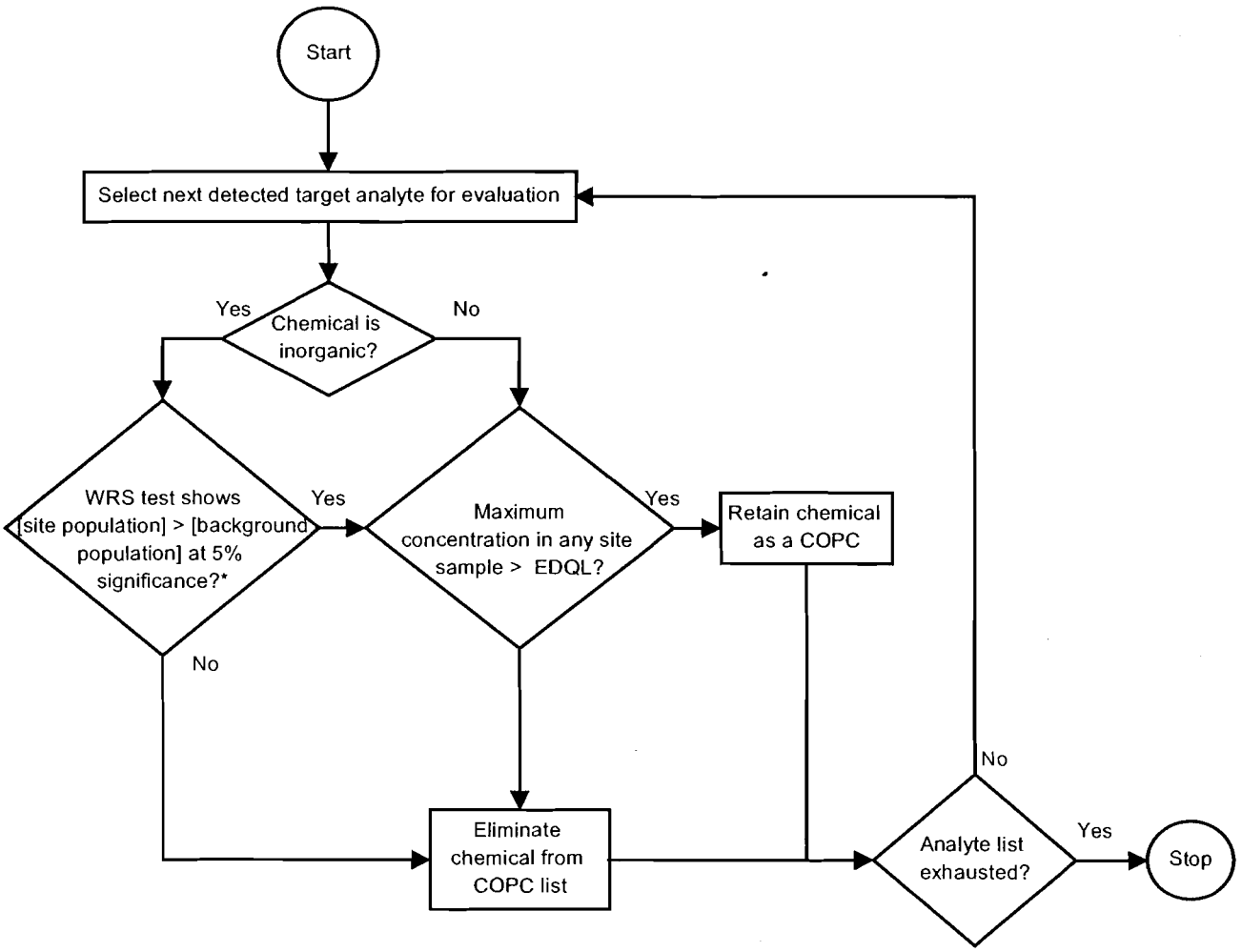
ECOLOGICAL RISK ASSESSMENT COPC SELECTION FOR SURFACE WATER AND SEDIMENT NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
EDQL = Ecological data quality level
WRS = Wilcoxon Rank Sum
*The upstream population will be represented by samples collected upstream of the SWMU investigative samples.

FIGURE 1-16

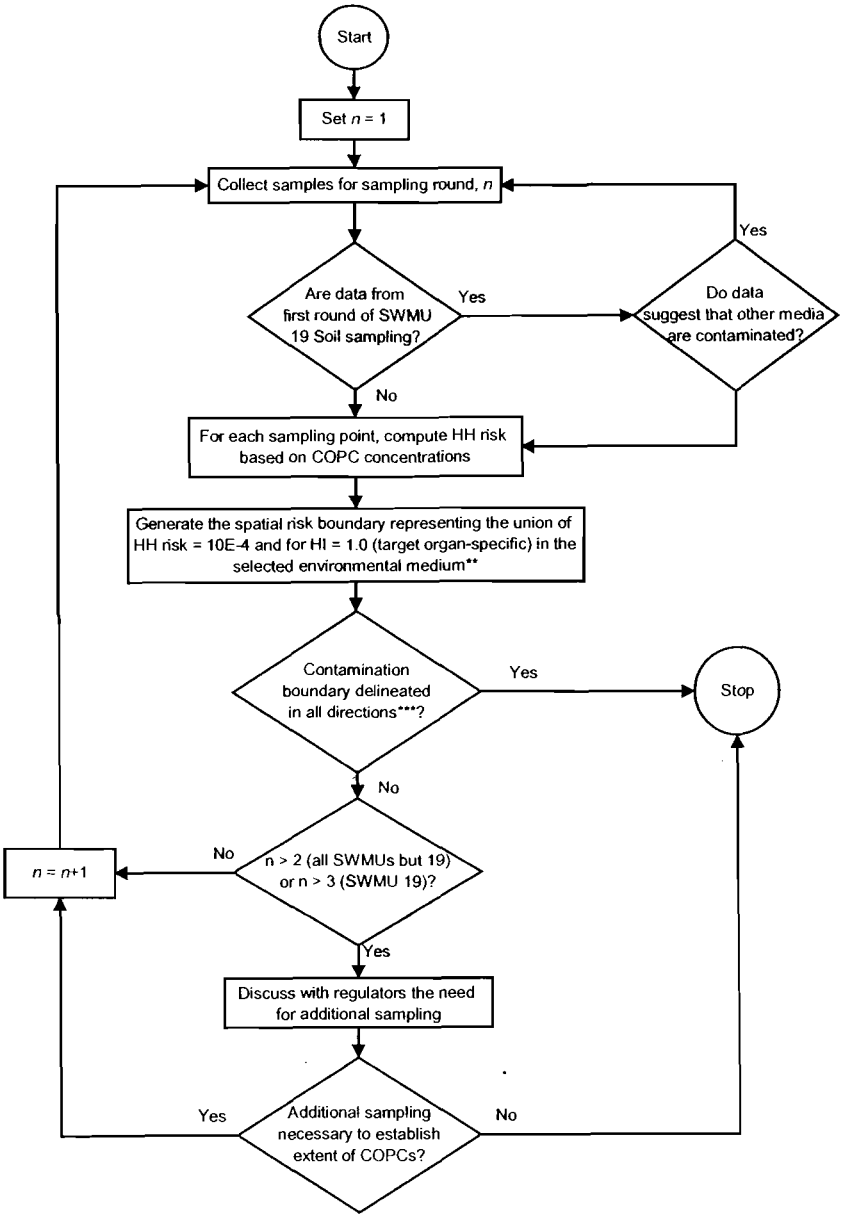
ECOLOGICAL RISK ASSESSMENT COPC SELECTION FOR SURFACE SOIL NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
EDQL = Ecological data quality level
WRS = Wilcoxon Rank Sum
*The background population will be represented by soil data from the NSWC Basewide Background Soil Investigation that most closely matches the site soil in terms of depositional environment, depth, and grain size. If multiple soil types are present at the site, multiple background soil types will be used, as necessary, to obtain a reasonable match for each site soil type.

FIGURE 1-17

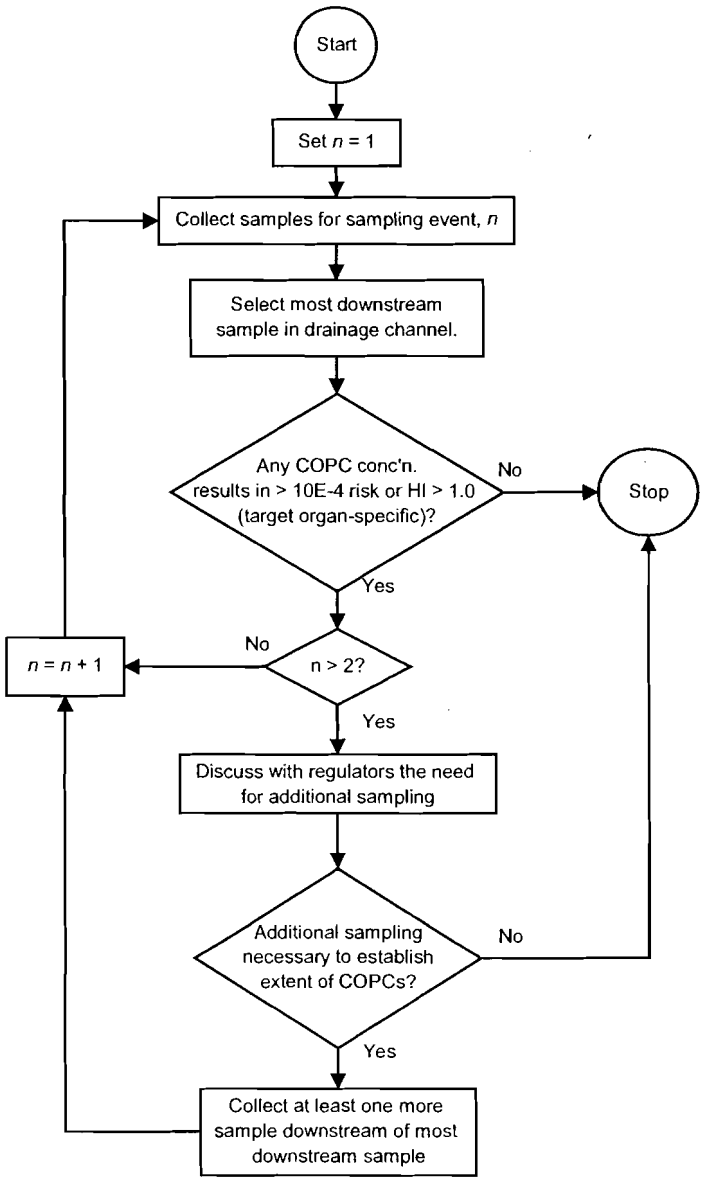
DECISION RULE FOR ESTABLISHING NATURE AND EXTENT OF COPCS IN GROUND WATER AND SOILS (SWMUS 12, 13, 16)* NSWC CRANE, CRANE, INDIANA



*This decision diagram will be applied to each environmental medium individually.
** For surface soils, the risk boundary will be a two-dimensional boundary based on COPC concentrations to a depth of 2 feet. For subsurface soils, the risk boundary will be a three-dimensional boundry based on [COPC]s at > 2 feet deep. For ground water, risk will be computed for each well location based on [COPC]s in the ground water.
***The spatial risk boundary representing 1E-4 human health (HH) cumulative cancer risk and the spatial boundary representing a hazard index (HI) of 1.0 will each be generated and plotted separately. Once plotted, the best fit boundary including both the spatial HH cumulative cancer risk boundary and spatial HI boundry will be generated to represent the union of 1.4E-4 HH cumulative cancer risk and HI=1.0.

FIGURE 1-18

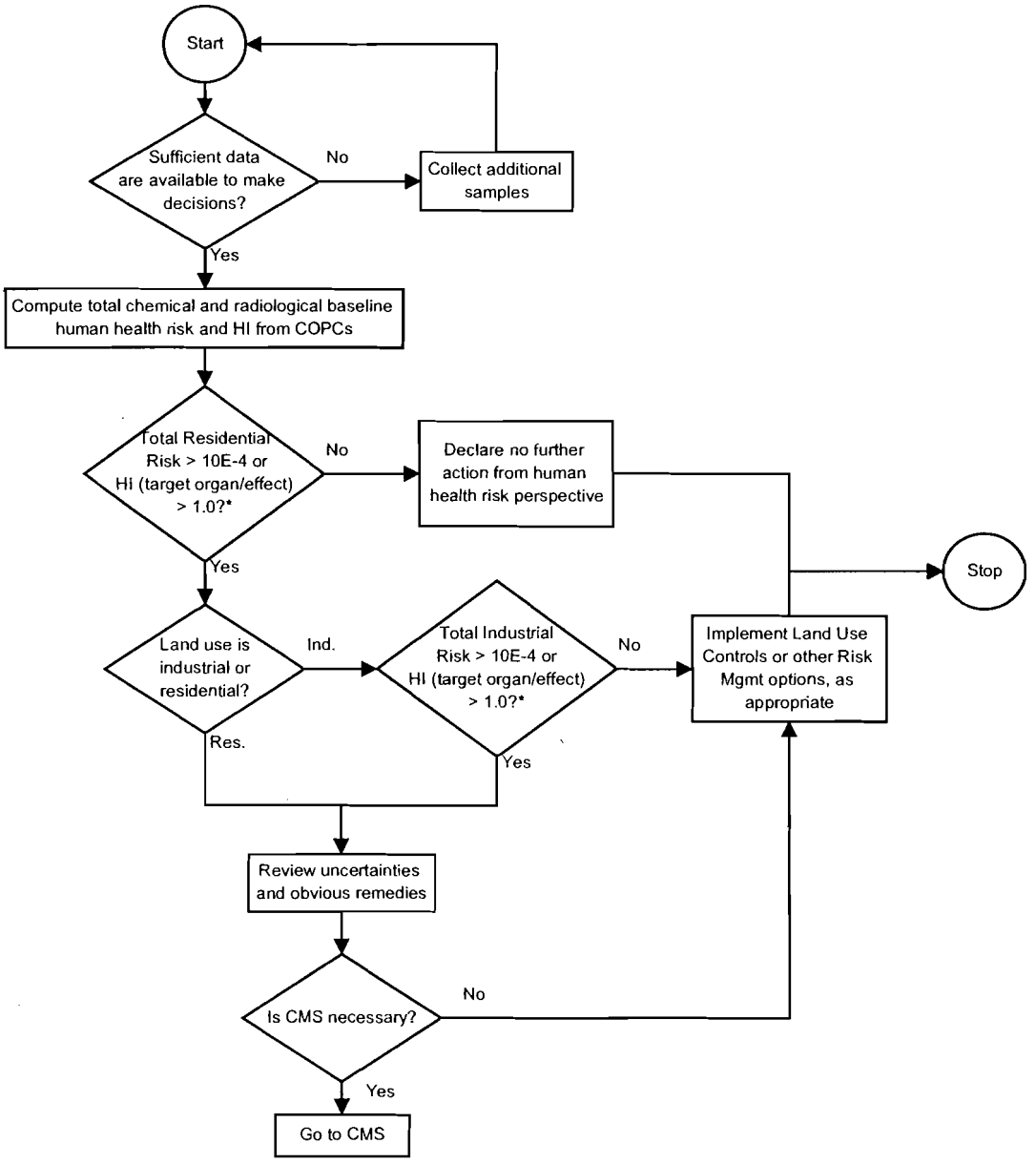
DECISION RULE FOR ESTABLISHING NATURE AND EXTENT OF COPCS IN SURFACE WATER AND SEDIMENT NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
HI = Hazard Index
n = Loop counter

FIGURE 1-19

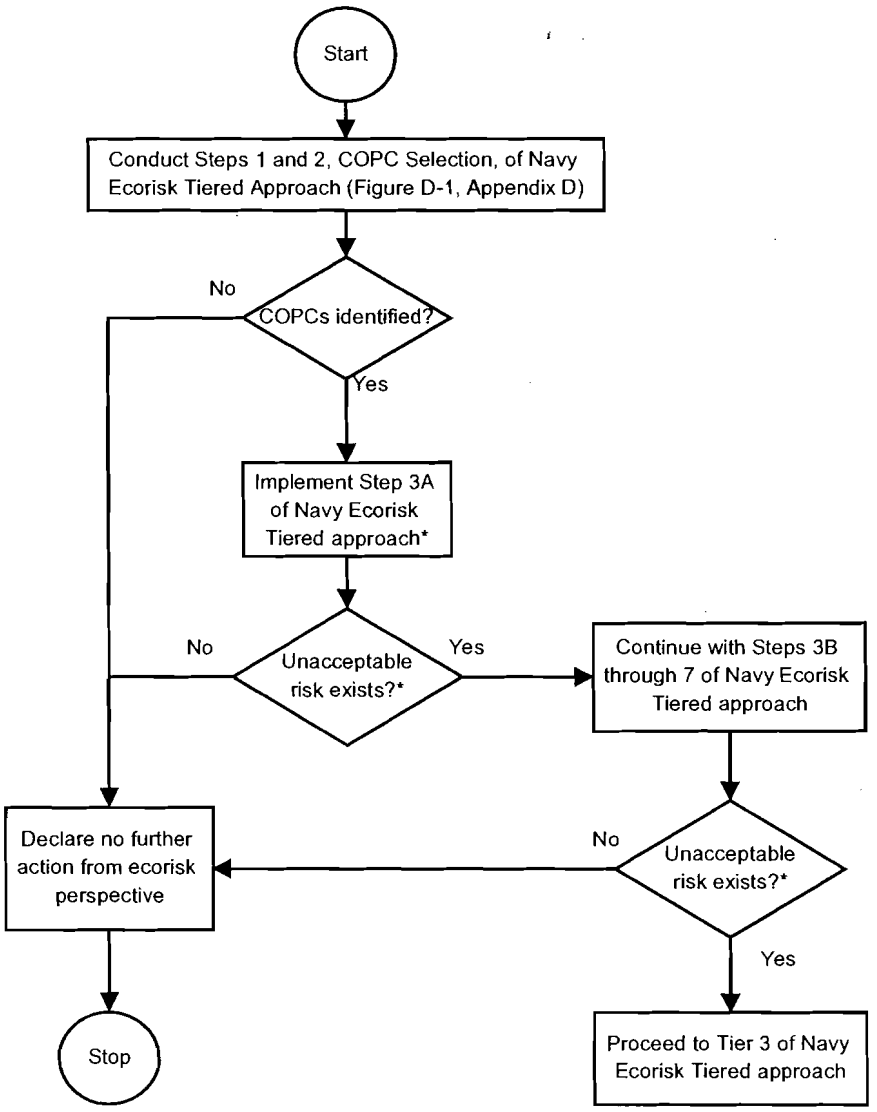
BASELINE HUMAN HEALTH RISK ASSESSMENT DECISION FLOW NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern
CMS = Corrective Measures Study
HI = Hazard Index
* The computed risk values will be based on the exposure point concentrations explained in the text, Section 1.4.4. Total risk is defined as total excess lifetime cancer risk (see 40CFR300.430(e)(2)(i)(A)(2) or excess non-cancer risk for the RME receptor. Risk estimates for naturally occurring inorganics (at background concentrations) are not included in the risk estimates but will be presented and discussed in the uncertainty section of the baseline risk assessment and will be reviewed with the regulatory members of the NSWC Crane team for concurrence.

FIGURE 1-20

SCREENING-LEVEL AND STEP 3A ECOLOGICAL RISK ASSESSMENT DECISION FLOW NSWC CRANE, CRANE, INDIANA



COPC = Chemical of potential concern

* This evaluation will include, but not be limited to, consideration of habitat, magnitude of risk-level exceedences, bioavailability of COPCs, and frequency of COPC detection

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section presents the project management and organization for this Phase III multimedia RFI at SWMUs 12, 13, 16, and 19 at NSWC Crane. Discussed in the following subsections are the staffing and coordination requirements.

2.1 MANAGEMENT

TtNUS, on behalf of the U.S. Navy, is responsible for the overall management, implementation of contract field activities, and preparation of the SWMUs 12, 13, 16, and 19 Phase III multimedia RFI QAPP. Personnel from the Navy will be actively involved and will coordinate with TtNUS personnel in a number of areas. The authorities and organizational relationships of key personnel are depicted on Figure 2-1. Corresponding addresses and telephone numbers of key personnel are listed by organization in Table 2-1. Responsibilities for program management, project management, field operations, and laboratory operations are discussed in the following sections. It is intended that the individuals named will perform the designated responsibilities to the extent that the specific person is available to perform the stated activities.

2.1.1 U.S. EPA Project Manager

The U.S. EPA project manager (PM), Mr. Peter Ramanauskas, will oversee the implementation of the SWMUs 12, 13, 16, and 19 Phase III multimedia RFI at NSWC Crane. The U.S. EPA PM represents the Agency's interests and will provide input from this perspective and lend general historical and technical assistance to NSWC Crane field activities.

2.1.2 Indiana Department of Environmental Management

The IDEM Hazardous Waste representatives, Mr. Doug Griffin and Mr. Marty Harmless, will oversee the implementation of this Phase III RFI investigation. They represent IDEM's interests and will provide input from this perspective.

2.1.3 Navy Project Managers

The Navy remedial project manager (RPM), Mr. Bill Gates, acts as the focal representative for the U.S. Navy, providing management, technical direction, and oversight for all NSWC Crane project activities performed by contractors (i.e., TtNUS) and their subcontractors. In matters such as facilitation of site

access, and oversight, etc., the Navy RPM is assisted by the NSWC environmental site manager (ESM), Mr. Tom Brent. Additional responsibilities of the RPM are as follows:

- Define project objectives and develop a detailed QAPP schedule.
- Establish project policy and procedures to address the specific needs of the project as a whole, as well as the objectives of each task.
- Acquire and apply technical resources (i.e., contractors) as needed to ensure performance within budget and schedule constraints.
- Review the work performed on each task to ensure its quality, responsiveness, and timeliness.
- Review and analyze overall task performance with respect to planned requirements and authorizations.
- Approve all reports (deliverables) before their submittal to U.S. EPA Region 5.
- Ultimately be responsible for the preparation and quality of interim and final reports.
- Represent the project team at meetings and public hearings.

2.1.4 Contractor Project Management

Program Manager

The TtNUS Navy Southern Division Comprehensive Long-Term Environmental Action Navy (CLEAN) program manager, Ms. Debbie Wroblewski, provides operations, technical, and administrative leadership and oversees and supports quality policies. The program manager assigns project task order managers (TOMs) and oversees their performance. The program manager also ensures the availability of technical and support resources for program operations and maintains consistency in procedures and projects among CTO assignments. In these matters, the program manager is assisted by the TOM.

Task Order Manager

The TtNUS TOM, Mr. Ralph Basinski, has overall responsibility for ensuring that the project meets U.S. EPA and IDEM objectives and Navy and TtNUS quality standards. The TOM is responsible for the preparation and distribution of the QAPP at the direction of the Navy RPM to all parties connected with the project, including any subcontractors. The TOM will report to the Navy RPM and is responsible for technical QC and project oversight. Additional responsibilities of the TOM are as follows:

- Ensure timely resolution of project-related technical, quality, safety, or waste management issues.
- Function as the primary interface with the Navy RPM and NSWC Crane ESM, field and office personnel, and subcontractor points-of-contact.
- Ensure that health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories.
- Monitor and evaluate subcontractor laboratory performance.
- Coordinate and oversee work performed by field and office technical staff (including data validation, statistical evaluations, and report preparation).
- Coordinate and oversee maintenance of all project records.
- Coordinate and oversee review of project deliverables.
- Prepare and issue final deliverables to the Navy.
- Approve the implementation of corrective actions.

Project Chemist

The TtNUS project chemist, Ms. Linda Karsonovich, has the overall responsibility for ensuring that the project meets objectives from the standpoint of laboratory performance. The project chemist is responsible for the technical preparation of laboratory statements of work (SOWs) and work releases. All

subcontractor laboratory project managers will report to the project chemist. The project chemist will report to the TOM. Additional responsibilities of the project chemist are

- Provide technical advice to the team on matters of project chemistry.
- Monitor and evaluate subcontractor laboratory performance.
- Ensure timely resolution of laboratory-related technical, quality, or other issues affecting project goals.
- Function as the primary interface with all subcontracted laboratories and the TOM.
- Coordinate and oversee work performed by all subcontracted laboratories.
- Coordinate and oversee review of laboratory deliverables.
- Recommend appropriate laboratory corrective actions.

Health and Safety Manager

The TtNUS health and safety manager (HSM), Mr. Matt Soltis, is responsible for the following:

- Provide technical advice to the TOM on matters of health and safety.
- Oversee the development and review of the HASP.
- Implement the HASP.
- Assign the site safety officer (SSO) and supervise his/her performance.
- Conduct health and safety audits.
- Prepare health and safety reports for management.

2.2 QUALITY ASSURANCE (QA)

This section identifies the QA responsibilities for this Phase III RFI. Responsibilities of U.S. EPA Region 5, TtNUS, and the analytical laboratories are discussed.

2.2.1 U.S. EPA Region 5 Quality Assurance Coordinator

The U.S. EPA Region 5 RCRA quality assurance coordinator (RQAC), Mr. Allen Debus, has the responsibility to review and approve the QAPP and to provide overall QA support and review. Additional responsibilities may include the following:

- Coordinate external performance and system audits of the contracted laboratory
- Review and evaluate analytical field and laboratory procedures

2.2.2 TtNUS QA Manager

The TtNUS quality assurance manager (QAM), Mr. Paul Frank, is responsible for overall QA for the project and reports directly to the TtNUS program manager. He acts on behalf of the U.S. Navy for project quality assurance. The QAM is responsible for the following:

- Develop, maintain, and monitor QA policies and procedures.
- Provide training to TtNUS staff in QA/QC policies and procedures.
- Conduct systems and performance audits to monitor compliance with environmental regulations, contractual requirements, QAPP requirements, and corporate policies and procedures.
- Audit project records.
- Monitor subcontractor quality controls and records.
- Assist in the development of corrective action plans and ensure correction of nonconformances reported in internal or external audits.
- Oversee the implementation of the QAPP.
- Oversee and review the development and revision of the QAPP.
- Oversee the responsibilities of the TtNUS site QA/QC advisor.
- Prepare QA reports for management.

2.2.3 TtNUS Project QA Chemist

The TtNUS project chemist, Ms. Linda Karsonovich, supports the TOM in preparing and reviewing the QAPP, coordinating work performed by office technical staff, and resolving matters concerning project chemistry. The project chemist also supports the project QA advisor on matters of QA/QC.

2.2.4 TtNUS Project QA Advisor

The TtNUS project QA advisor, Dr. Tom Johnston, supports the TOM in preparing and reviewing the QAPP and conducting data assessments. The Project QA Advisor communicates directly with the QAM on matters of QA/QC.

2.2.5 TtNUS Data Validation Manager

The TtNUS data validation manager will be responsible for scheduling completion of the reduction, validation, and documentation activities related to the sample data package deliverables received from the laboratories. The data validation manager will report to the TOM or designee.

2.2.6 Laboratory Responsibilities

Laucks Testing Laboratories, Inc., of Seattle, Washington, will perform all sample analyses for this field activity, with the exception of dioxin analysis and dye analysis. Dioxin analysis will be performed by Triangle Laboratories as a subcontractor to Laucks Testing. Dye analysis will be performed by the NSWC Crane Laboratory, which has specifically developed analytical methods for the Crane site.

The subcontracted laboratories are responsible for analyzing all samples in accordance with the analytical methods and additional requirements specified in the attendant QAPP. It also will be the analytical laboratory's responsibility to properly dispose of unused sample aliquots. Responsibilities of key laboratory personnel are outlined in the following paragraphs.

Laboratory Project Manager

The laboratory project manager will interface directly with the TtNUS TOM and QA Advisor and will perform the following:

- Ensure that method- and project-specific requirements are properly communicated and understood by laboratory personnel.
- Ensure that all laboratory resources are available on an as-required basis.
- Monitor analytical and project QA requirements.
- Review data packages for completeness, clarity, and compliance with project requirements .
- Inform the TtNUS TOM of project status and any sample receipt or analytical problems.

Laboratory Operations Manager

Responsibilities of the laboratory operations manager include the following:

- Support the QA program within the laboratory.
- Provide management overview of both production- and quality-related laboratory activities.
- Maintain adequate staffing to meet project analytical and quality objectives.
- Approve all laboratory SOPs and QA documents.
- Supervise in-house chain-of-custody documentation.
- Oversee the preparation of, and approval of, final analytical reports before submittal to TtNUS.

Laboratory Quality Assurance Officer (QAO)

The laboratory QAO will report directly to the laboratory operations manager. The laboratory QAO will be independent of laboratory production management to ensure that laboratory quality performance is assessed without schedule and cost considerations. Responsibilities of the laboratory QAO include the following:

- Define appropriate laboratory QA procedures and monitor overall laboratory QA.
- Stop work if a condition adverse to the quality of work is encountered, if QA or QC procedures are not followed, or if analytical out-of-control events are encountered that have not been corrected.
- Approve and maintain document control of all QA documents and SOPs.
- Perform and/or implement internal system and performance audits and verify completion of corrective actions cited in audits.
- Direct laboratory participation in laboratory accreditation and certification programs.

Laboratory Sample Custodian

The laboratory sample custodians will report to the laboratory operations manager. Responsibilities of the laboratory sample custodian include the following:

- Receive and inspect the incoming sample containers.
- Record the condition of the incoming sample containers.
- Sign appropriate documents.
- Verify chain-of-custody.
- Notify laboratory project manager of sample receipt and inspection.
- Assign a unique identification number and customer number, and enter each into the sample receiving log.
- With the help of the laboratory project manager, initiate transfer of the samples to appropriate lab sections.
- Control and monitor access and storage of samples and extracts.

Laboratory Technical Staff

The laboratory technical staff will be responsible for sample analysis based on the analytical methods and requirements specified in the attendant QAPP.

2.3 FIELD INVESTIGATION

TtNUS will be responsible for all field activities related to this Phase III RFI. The TtNUS field team will be organized according to the activities planned. Field team members will be selected based on the type and extent of effort required. All team members will be appropriately skilled and trained for the tasks they are assigned to perform. The team will consist of a combination of the following personnel:

- Field operations leader (FOL)
- Site QA/QC advisor
- SSO
- Site UXO specialist
- Field technical staff

2.3.1 Field Operations Leader

The FOL is responsible for coordinating all on-site personnel and for providing technical assistance, when required. The FOL, or designee, will coordinate and lead all sampling activities and will ensure the availability and maintenance of all sampling materials and equipment. The FOL is responsible for completing all sampling, field, and chain-of-custody documentation, will assume custody of all samples, and will ensure the proper handling and shipment of samples. The FOL will report directly to the TtNUS TOM. Specific FOL responsibilities include the following:

- Ensure that all health and safety requirements unique to this site are implemented.
- Function as the communications link between field staff members, SSO, UXO specialist, the NSWC Crane Environmental site manager, and the TOM.
- Alert off-site analytical laboratories of any special health and safety hazards associated with environmental samples.
- Oversee the mobilization and demobilization of all field equipment and subcontractors.
- Coordinate and manage the field technical staff.
- Adhere to the work schedules provided by the TOM.
- Ensure the proper maintenance of the site logbook, field logbook, and field recordkeeping.
- Initiate field task modification requests when necessary.
- Identify and resolve problems in the field, resolve difficulties via consultation with the NSWC Crane environmental site manager, implement and document corrective action procedures, and provide communication between the field team and project management.

2.3.2 Site QA/QC Advisor

The FOL (or designee) will act as the site QA/QC advisor and will be responsible for ensuring adherence to all QA/QC requirements as defined in the QAPP. Strict adherence to these procedures is critical to the collection of acceptable and representative data. The following is a summary of the Site QA/QC Advisor's responsibilities:

- Ensure that field QC samples are collected at the proper frequency.
- Ensure that additional volumes of sample are supplied to each analytical laboratory with the proper frequencies to accommodate laboratory QA/QC analyses.
- Ensure that measuring and test equipment are calibrated, used, and maintained in accordance with applicable procedures and technical standards.
- Act as liaison among site personnel, laboratory personnel, and the QAM.
- Manage bottleware shipments and oversee field preservation.

2.3.3 Site Safety Officer

The FOL (or designee) will also serve as the SSO. The duties of the SSO are detailed in the Health and Safety Plan (HASP). The SSO has stop-work authority, which can be executed upon the determination of an imminent safety hazard.

2.3.4 Site UXO Specialist

Based on the potential for UXO hazards associated with this investigation, a TtNUS UXO specialist will be on site at all times that invasive activities are being conducted at SWMUs 12, 13, 16, and 19. The duties of the UXO specialist are detailed in the HASP. The UXO specialist has stop-work authority, which can be executed upon the determination of an imminent UXO safety hazard.

2.3.5 Field Technical Staff

The field technical staff for this project will be drawn from TtNUS's pool of qualified personnel. All the designated field team members will be experienced professionals who possess the degree of specialization and technical competence required to effectively and efficiently perform the required work.

Field staff are responsible for complying with field-related requirements as presented in the QAPP and the HASP.

2.4 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATIONS

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in health and safety training, as described under Occupational Safety and Health Administration (OSHA) 29 CFR 1910.120(b)(4). The UXO specialist will be certified in accordance with TtNUS SOP HS-2.0, which is appended to the QAPP.

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TABLE 2-1

**KEY PROJECT PERSONNEL
NAMES, PHONE NUMBERS AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 3**

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
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TABLE 2-1

KEY PROJECT PERSONNEL
NAMES, PHONE NUMBERS AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 3

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
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Kathy Krepps Lab Operations Manager Laucks Testing	Laucks Testing 940 South Harney Street Seattle, WA 98108	Phone: (206) 767-5060 FAX: (206)767-5063

TABLE 2-1

**KEY PROJECT PERSONNEL
NAMES, PHONE NUMBERS AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 3 OF 3**

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
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Eric Wendland Project Manager APCL	Applied Physics and Chemistry Lab 13760 Magnolia Avenue Chino, CA 91710	Phone: (909) 590-1828 FAX: (909) 590-1498

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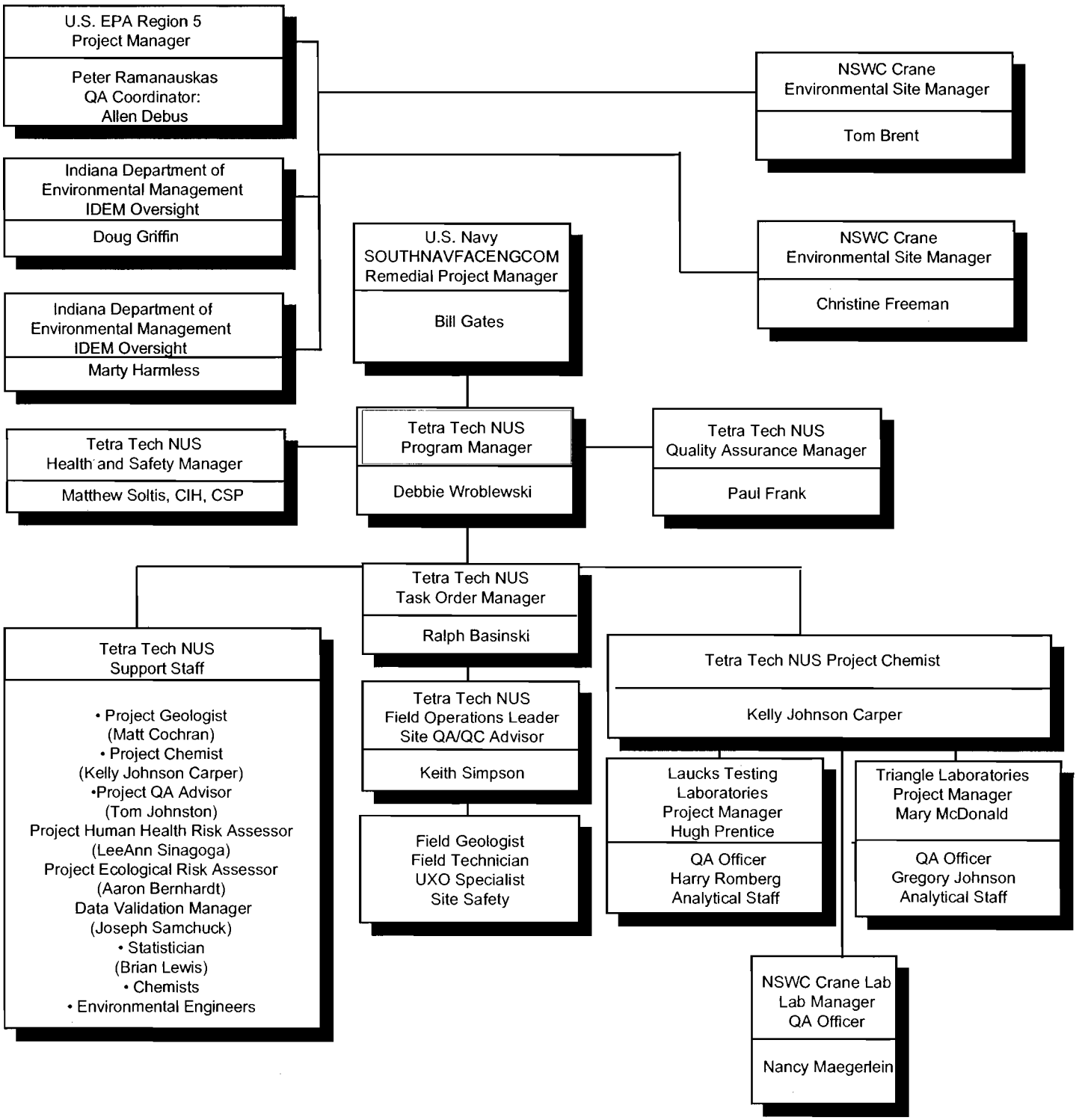
FIGURE 2-1

PROJECT ORGANIZATION CHART
SWMUs 12, 13, 16, AND 19
NSWC CRANE, CRANE, INDIANA



3.0 SAMPLING AND ANALYSIS PLAN

3.1 GENERAL APPROACH

This chapter describes sampling locations and rationales for the sampling activities and the equipment and procedures to be used for collecting, handling, preserving, and shipping the samples to the analytical laboratories. The text references SOPs (included in Appendix H) and the HASP, when applicable.

Prior to sampling, the TOM will ensure that all field personnel read and understand the QAPP and HASP, the FOL will ensure that all required field equipment for non-health-and-safety operations is available and operational, and the SSO will ensure that all health-and-safety-related equipment is available and operational.

The following four SWMUs will be investigated at NSWC Crane during this project:

- Mine Fill A (SWMU 12/14)
- Mine Fill B (SWMU 13/14)
- Cast High Explosives Fill (SWMU 16/16)
- Pyrotechnic Test Areas (SWMU 19/00)

The overall sampling strategy for this investigation took into consideration several factors. Those factors included:

- A desire to delineate contaminated areas so that risk to human and ecological receptors could be bounded spatially.

- A desire to evaluate realistic risks incurred by human and ecological receptors.

- A desire to evaluate the impact of soil contamination, if any, to ground water.

- In some cases, a desire to determine whether any chemical releases have occurred at particular locations.

The initial sampling locations at all four SWMUs are generally biased toward areas that are likely to be contaminant source areas or areas that are transport conduits (e.g., drainage ditches) for contamination.

This sampling strategy is likely to overestimate risks to human and ecological receptors because the observed chemical concentrations are likely to be greater than those to which receptors are actually exposed. If risk estimates based on the initial round of samples indicate the presence of an unacceptable risk at a SWMU, additional sampling may be proposed at that SWMU to obtain a more realistic estimate of risks incurred by human and ecological receptors through use of a more representative sampling strategy. For that reason, the decision was made to allow for multiple rounds of sampling, as indicated in Figures 1-17 through 1-19. If additional samples are collected, it is likely that a statistically based sampling approach will be used.

Sampling of all media is planned for the Mine Fill A and Mine Fill B areas. However, soil sampling will be limited to those potential source areas that have not yet been investigated or that have detected contamination that requires further characterization or to provide information on soil properties. Extensive investigation and remediation of explosives-contaminated soils have been performed at Mine Fills A and B during previous efforts. It is anticipated that the soil data collected during those activities will be used to augment the data collected from this effort to provide a comprehensive evaluation of Mine Fills A and B.

All media will be sampled at the Cast High Explosives Fill during this investigation. Previous investigations including multimedia sampling have been performed at this SWMU, and contamination has been detected in several of the media. The data from these investigations will also be used to supplement the data collected from this effort during the evaluation of this SWMU.

Mine Fills A and B and the Cast High Explosives Fill are located on topographic high points, and radial surface water runoff is anticipated based on an evaluation of topography and the locations of drainage ditches at each SWMU. The potential for ground water to flow in a radial direction also exists at each of these SWMUs. Therefore, perimeter sampling will be performed along all borders to address the off-site impacts from SWMU activities. Source sampling will also be performed at each SWMU where additional spatial coverage is needed or where potential hotspot areas exist that would not be adequately addressed with the perimeter sampling.

No sampling data exist for the Pyrotechnic Test Area. This SWMU consists of multiple potential surface source areas covering a large area within a flood plain. However, it is not known which, if any, of these potential source areas are contaminated. Therefore, a multi-round sampling approach will be employed at this SWMU. Each round will consist of data collection, evaluation of the data results, and recommendation, consultation, and concurrence for additional sampling rounds with U.S. EPA Region 5. This QAPP addresses all the necessary components for an initial sampling round at this SWMU, including the collection of surface and subsurface soil samples in the likely source areas, as well as sampling of

surface water and sediment in the drainage ditches and creeks. Sample locations and numbers for this initial sampling round are also identified in this QAPP. The intention of the initial sample round is to determine whether contamination is present or absent at the likely potential source areas.

A second round of sampling, consisting of additional surface and subsurface soil sampling, may be performed at those areas of SWMU 19 where contamination has been identified during the initial sampling round, with the intention of determining the extent of soil contamination at each source area. In addition, a third sampling round to include monitoring well installations will be performed in those areas where soil contamination has been identified during Rounds 1 and 2 at levels that may impact ground water. The intention of the third sampling round will be to determine whether ground water contamination exists in those areas of interest and, if possible, to determine the nature and extent of the ground water contamination. The exact numbers and locations of sampling points for the second and third sampling rounds are unknown at this time; however, a maximum number of sample points has been estimated for discussion purposes. The intention of this QAPP will be to address all the necessary components for Rounds 2 and 3 with the exception of sample locations, actual numbers, depths, and rationale for the samples collected. This information will be provided to the U.S. EPA Region 5 between each sample round for discussion and concurrence via meetings, conference calls, and correspondence and will be documented for the administrative record. This phased sampling should assure decision makers of progress toward attaining project objectives and should afford the earliest opportunity for declaring attainment of those objectives. Additional sampling stages that may be required beyond this initial stage are expected to involve limited sampling and analyses by taking advantage of knowledge gained during the previous sampling rounds.

The sampling to be performed as part of this field effort will include surface soil, subsurface soil, ground water, surface water, and sediment with a wide variety of analyses. The types of analyses to be performed on these samples are wide ranging and commensurate with what is known and suspected about the types or quantities of chemicals that were present or may have been present at each of the SWMUs. Analytes to be evaluated in each environmental medium are listed in Tables 1-3 (SWMUs 12 and 13), 1-4 (SWMU 16), and 1-5 (SWMU 19).

3.2 SAMPLE IDENTIFICATION SYSTEM

All samples will be properly labeled with an adhesive-backed sample label affixed to, and a tag tied to, each sample container in accordance with SOP CTO166-01. The sample labels and tags will include the following information: project name, project location, sample tracking number, sampling date, sampling

time, type of analysis required, matrix type, preservative, initials of sampler, and the name of the analytical laboratory to which the sample will be submitted.

Each collected sample will be assigned a unique sample tracking number. The sample tracking number for soil and sediment samples is a four-segment, alphanumeric code beginning with the site identification (12,13,16, or 19 represents the SWMU number) and followed by codes for the sample type, sample location, and sample depth. The sample tracking number for the other media is a five-segment, alphanumeric code identifying the site ID, sample type, sample location, sampling round, and filtering indicator. These numbering schemes are described in SOP CTO166-02. Any other pertinent information regarding sample identification will be recorded in the field logbooks and sample log sheets.

The sampling time recorded on the chain-of-custody form and labels for duplicate samples will be 0000 so that the duplicate samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

Matrix spike and matrix spike duplicate (MS/MSD) samples will be designated on the field documentation forms and chain-of-custody form (see SOP CTO166-03).

3.3 UNEXPLODED ORDNANCE (UXO) AVOIDANCE

The activities performed at each of the SWMUs, which included ordnance testing and manufacture, were performed using good housekeeping practices. The potential for encountering buried UXO is low, and borings will be located in areas with the least likelihood of encountering UXO while still maintaining project objectives. However, specific boring locations as identified by NSWC may require screening using a magnetometer the presence of UXO in accordance with the HASP. Prior to the initiation of intrusive site activities a meeting will be held with NSWC Crane staff to determine whether any boring locations may require UXO screening. If during investigative activities potential ordnance is encountered, activities will stop and the Crane Environmental Site Manager will be notified.

UXO screening will not be conducted at SWMUs 12, 13, or 16. SWMUs 12 and 13 only granular explosives were released, and soils contaminated with explosives have been remediated. At SWMU 16 no ordnance items were managed on the ground. Released materials consisted only of ash on slag. UXO screening is conducted with a magnetometer which is sensitive to magnetic material. Explosives are not magnetic and would not be detected by a magnetometer. Visual inspection will be used to detect bulk explosives.

All measurements made with the field instruments (listed in Table 3-1) and visual observations concerning the subsurface materials encountered will be recorded on boring log sheets. Any encounters with metallic objects or discolored soil materials and any above-background measurements of VOCs will be immediately reported to the FOL and the SSO, and appropriate actions will be taken as specified in the HASP.

3.4 SAMPLING LOCATIONS, ANALYSES, AND RATIONALES

This section presents sampling locations, QA samples to be collected, analyses to be performed, and rationales for the sampling and analytical program. Details regarding the equipment and procedures for collecting, preserving, packaging, and shipping the samples are included in Section 3.7. Sampling locations are shown on Figures 3-1 through 3-4.

3.4.1 Surface and Subsurface Soils

Surface and subsurface soil samples will be collected at each of the SWMUs as described in the remainder of this section. The analyses to be performed on each soil sample are listed in Table 3-2. The rationales for parameter selection is discussed in Section 1.4.1 and detailed on Tables 1-3, 1-4, and 1-5.

Surface and subsurface soil sampling will be performed using direct-push technology (DPT) soil sampling or hollow-stem auger drilling with split-spoon sampling. Split-spoon sampling will be used only if DPT soil sampling does not give good recovery. All soil samples for VOC analyses will be collected with an Encore™ sampler immediately upon opening the split spoon or DPT sampler. Details regarding soil sampling equipment and procedures are included in Section 3.5.2 and SOP CTO166-04.

QA/QC samples will be collected at frequencies listed in Table 3-3. Duplicate samples and rinsate blanks will be collected at a rate of one for every 10 regular soil samples and analyzed for the same analytes as the corresponding soil samples. MS/MSD samples will be collected and analyzed in the fixed laboratory at a rate of one per every 20 soil samples. A trip blank will be placed in every cooler containing samples destined for VOC analyses. The total number of soil analyses for each analyte group is tabulated in Table 3-3.

3.4.1.1 Mine Fill A Subsurface Soil Sampling

As previously discussed, investigation and remediation have been performed at Mine Fill A as part of the installation of a bioremediation facility. However, no soil sampling has been performed in the vicinity of a

conical-shaped asphalt pad that was observed during a site visit performed by TtNUS in June 2001. Therefore, one soil boring (12SB01) is proposed for this area (immediately adjacent to the pad on the downslope edge along a ditch) to determine whether any soil contamination is present as a result of activities performed at this pad. The boring will be sampled at two depth intervals, including one sample 0 to 2 feet below ground surface (bgs) and a second sample immediately above the water table.

Two soil borings (12SB02 and 12SB03) will be installed and sampled to establish soil properties (total organic carbon, cation exchange capacity, and pH) for the soil replacement areas that are part of the bioremediation facility. Each boring will be sampled at two depth intervals, including one sample at 0 to 2 feet bgs and a second sample immediately above the water table.

Four soil borings each will be installed along the edges of settling basins located at structures 3110 (12SB04 through 12SB07) and 3037 (12SB08 through 12SB11) to evaluate whether leakage may have occurred. Each boring will be sampled at one surface and two subsurface depth intervals above the water table; specific subsurface depths will be determined in the field based on encountering waste or obvious contamination during drilling. If no waste or obvious contamination is encountered during drilling, then the shallow subsurface sample will be collected from a depth that is representative of the middle depth of the settling basin. The deep subsurface sample will be collected from a depth that is representative of the bottom of the settling basin, but not below the water table.

3.4.1.2 Mine Fill B Surface and Subsurface Soil Sampling

As previously discussed in Section 1, PCB soil contamination exists in the vicinity of Buildings 166 and 171 as a result of leaking Therminol boilers. The boilers and soil have been removed, but PCBs at concentrations greater that 10 mg/kg are still believed to exist in these areas. Therefore, 14 additional soil borings (13SB01 through 13SB06 and 13SB09 through 13SB16) are proposed to be installed immediately outside the known areas of soil removal to determine whether any residual PCBs are present. One surface soil sample (0 to 2 feet bgs) and one subsurface soil sample (2 to 4 feet bgs) will be collected from each boring.

Two soil borings (13SB07 and 13SB08) will be installed and sampled to establish soil properties (total organic carbon, cation exchange capacity, and pH) for the soil replacement areas as part of the biotreatment facility. Each boring will be evaluated over two depth intervals, including one sample at 0 to 2 feet bgs and a second sample immediately above the water table.

3.4.1.3 Cast High Explosives Fill Surface and Subsurface Soil Sampling

As previously discussed in Section 1, soil contamination exists in the incineration ash disposal areas south of Building 146, and shell casings are present on the ground surface in the parking areas surrounding Building 146. Some of the ash has been removed; however, contamination may still be present. Therefore, 39 additional soil borings (16SB01 through 16SB39) are proposed to be installed within and immediately outside these areas to determine whether any residual contamination is present. One surface soil sample (0 to 2 feet bgs) and one subsurface soil sample (depth to be determined in the field based on encountering waste or obvious contamination during drilling) will be collected from each boring. If no waste or obvious contamination is encountered during drilling, then the subsurface soil sample will be collected immediately above the water table.

Two soil borings (16SB40 and 16SB41) will be installed along the downgradient edges of sumps that previously conveyed water containing chlorinated constituents into ditches east and west, away from Building 146. Each boring will be evaluated over two depth intervals above the water table; specific depths will be determined in the field based on encountering waste or obvious contamination during drilling. The shallow soil sample will be collected from a depth that is representative of the middle depth of each sump and the deep sample will be collected from a depth that is representative of the bottom of each sump, but not below the water table.

3.4.1.4 Pyrotechnic Test Areas Surface and Subsurface Soil Sampling

The majority of testing operations at the Pyrotechnic Test Area consisted of controlled above-ground operations, with releases of contamination to the surface soil as the result of fallout. In some of the areas, testing of rockets or bombs would have resulted in both surface and shallow subsurface soil contamination, and below-ground operations may have resulted in subsurface soil contamination.

The proposed Round 1 surface soil sampling approach at this SWMU consists of locating 71 surface soil samples from areas where the greatest potential for surface soil contamination exists; these areas are the treatment areas at this SWMU. Samples 19SS01 through 19SS57 and 19SS67 through 19SS71 are intended to address potential contamination in proximity to source areas at the Ordnance Test Area and Annex. Samples 19SS50 through 19SS57 are intended to address the potential for widespread transport of surface contamination throughout the floodplain during flooding conditions (samples 19SS56 and 19SS57 can also address fallout along the warhead testing trajectory and deposition of contaminants from smokes). Samples 19SS58 through 19SS66 will address the rocket range source areas and rocket trajectory.

The number of samples proposed for each treatment area ranges from one for small isolated areas to six in larger treatment areas. The surface soil samples will be collected from a depth of 0 to 2 feet bgs.

Both surface and subsurface soil samples will be collected during Round 1 from soil borings at seven locations (19SB01 through 19SB07). One of the soil borings (19SB01) will be located in the vicinity of the fast cook-off tank where burnable fuel is pumped underground from a remote tank to the cook-off tank, then ignited. Soil borings 19SB02 and 19SB07 will be located adjacent to an underground brine tank (not identified on Figure 3-4) and saltwater wave tank, respectively, that are used to test underwater signal flares. The subsurface soil samples from these borings will be collected at a depth representing the tank bottoms, but not below the water table. Soil borings 19SB03 through 19SB06 will be located in a sand trap area that was the leading area for the rocket range.

Up to 120 additional surface soil samples and up to 60 soil borings for collection of subsurface soil samples may be installed during Round 2 to address those areas where contamination was encountered during Round 1. The exact locations, numbers, and depths will be determined after the Round 1 sample results are obtained and discussed with U.S. EPA Region 5.

3.4.2 Ground Water

Ground water will be evaluated during Round 1 at all the SWMUs with the exception of SWMU 19, which will be addressed during Round 3 if necessary. Existing monitoring wells are present only at SWMU 16.

The general objective of the ground water investigation at each of the SWMUs will be to determine whether ground water contamination is present and, if present, to define the magnitude and downgradient extent of shallow ground water contamination migrating from the site, as well the vertical extent of ground water contamination. Additional monitoring wells may be required during subsequent investigative rounds to fully meet the objective; however, the proposed approach is considered a reasonable effort based on the limited information available for each SWMU. Data collected during the initial round of ground water investigation at each SWMU will be presented to the U.S. EPA Region 5 with recommendations for any future work that is necessary to meet the project objectives. Monitoring wells will be installed initially to shallow depths in relative proximity downgradient of potential source areas, whereas subsequent installation rounds (depending on the presence of ground water contamination) may include shallow installations farther downgradient and deeper, to define contaminant extent.

The total depths and screen intervals anticipated for each new monitoring well will be determined in the field. However, screen lengths will be approximately 10 feet. Based on observations and information gathered during the drilling of each hole, the total depth of the hole and the placement of the well screen may be adjusted at the discretion of the field geologist or the FOL. Decisions concerning the monitored intervals and well depths will be based on the following (and possibly other) information collected while the well bore is being drilled and logged:

- The specific depths where the initial (shallow) and deeper water yielding zones are encountered during drilling.
- The specific depths where above-average rates of ground water yield are observed during drilling.
- The specific depth interval where contaminants (i.e., VOCs), if any, are noted during drilling.

All this information will be recorded on the borehole log as the hole is drilled.

Table 3-4 lists the ground water samples and the analyses that will be performed on each sample. One round of ground water samples will be collected from each of the wells. During low-flow well sampling, the temperature, specific conductance, pH, dissolved oxygen, ORP, and turbidity of the ground water will be measured and recorded for each well. If the turbidity of the well water samples cannot be reduced below 10 nephelometric turbidity units (NTUs) during low-flow sampling, then an aliquot of water will be filtered (see SOP CTO166-05) and analyzed for dissolved metals in addition to total metals.

The total number of ground water samples to be analyzed for each analyte group is listed in Table 3-5. The numbers of field QA/QC samples to be collected and analyzed are also listed in Table 3-5. One duplicate sample will be collected for every 10 ground water samples. Note: The duplicate samples will be preferentially collected from monitoring wells that appear to be contaminated (based on visual evidence, odor, or screening data). This will provide the greatest opportunity for computing a precision estimate with detectable analyte concentrations. One MS/MSD sample will be collected and analyzed for every 20 ground water samples analyzed. One rinsate sample will be collected and analyzed each day of ground water sampling activities. It is estimated that sampling will occur over four days. A trip blank will be included with each cooler containing samples for VOC analysis. It is estimated that four coolers will be necessary. Two source water blanks (tap water and distilled water) will be analyzed to establish whether contaminants may be present in the water used for steam cleaning and decontamination of sampling equipment.

3.4.2.1 Mine Fill A Ground Water Sampling

A total of 26 monitoring wells (12MWT01 through 12MWT26) will be installed at Mine Fill A. Mine Fill A is located on a topographic high, and it is anticipated that shallow ground water flow will be in a radial pattern away from the site toward topographic low areas. It is not known which well(s) (if any) will serve as an appropriate background well until a comprehensive round of water-level measurements is collected and a potentiometric surface map is prepared for this SMWU. The wells are located immediately downgradient of structures where historical soil contamination has been detected, as well as along the perimeter to provide adequate spatial coverage. Several wells (12MWT04, 12TMWT09, 12MWT14, 12MWT18, and 12MWT23) are located to provide adequate coverage within the interior areas of the SWMU. Both overburden and bedrock wells will most likely be installed at this site because it is possible that bedrock will be reached before the shallow ground water is encountered beneath the site. In this case, wells will be completed as bedrock installations. Otherwise, wells will be completed in the overburden. A list of the wells is included on Table 3-6.

3.4.2.2 Mine Fill B Ground Water Sampling

The approach for the ground water investigation at Mine Fill B is similar to Mine Fill A because they are similar in physical characteristics and operation. A total of 26 monitoring wells (13MWT01 through 13MWT26) will be installed at Mine Fill B. All wells are located along the SWMU perimeter with the exception of wells 13MWT04, 13MWT08, 13MWT10, 13MWT15, and 13MWT18, which are located in the interior of the SWMU. It is not known which well(s) (if any) will serve as an appropriate background well until a comprehensive round of water-level measurements is collected and a potentiometric surface map is prepared for this SMWU. The wells at Mine Fill B will be installed in the same manner as the wells in Mine Fill A. A list of the wells is included on Table 3-7.

3.4.2.3 Cast High Explosives Fill Ground Water Sampling

Five wells are currently located at the Cast High Explosives Fill. Information on these wells is included on Table 3-8 and in Appendix A. An additional five monitoring wells (16MWT01 through 16MWT05) will be installed to complement this existing monitoring well network because of the expectation that radial flow exists at this site. It is acknowledged that the predominant ground water flow direction is toward the south based on the existing well network (see Figure 1-6). However, it is believed that east and west components of flow may be identified once wells are installed in the vicinity of the two sumps at the SWMU. The two sumps are located adjacent to hillsides sloping to the east and west. It is not known which well(s) (if any) will serve as an appropriate background well until a comprehensive round of water-level measurements is collected and a potentiometric surface map is prepared for this SMWU. It is

anticipated that these wells will be completed as bedrock installations. A list of the wells is included on Table 3-9.

3.4.2.4 Pyrotechnic Test Areas Ground Water Sampling

Up to 30 monitoring wells may be installed during Round 3 to address those areas where soil contamination has been identified that could impact ground water. The exact locations, numbers, and depths will be determined after the Rounds 1 and 2 sample results are obtained. U.S. EPA Region 5 will be consulted for concurrence prior to installation of any monitoring wells. A list of the wells is included on Table 3-10.

3.4.3 Surface Water / Sediment

Surface water and sediment sampling is proposed for each of the four SWMUs. It is acknowledged that surface water may not be present at some of the sampling locations, depending on flow conditions at the time of sampling. The surface water samples will be paired with the sediment sample locations planned for the field effort. Surface water and sediment samples will be collected using the methods described in Sections 3.5.10 and 3.5.11.

Each surface water sample will be analyzed for the analyte groups listed in Table 3-11. Comparisons between upstream and downstream samples will only be made with the samples collected at the Pyrotechnic Test Areas, because the origin of surface waters at each of the other three SWMUs is within the SWMU interior.

Surface water QA/QC samples will be collected as detailed in Table 3-12. No rinsate blanks will be collected because the sample bottles will be filled directly, as discussed in Section 3.5.11. The field duplicate will be analyzed for all parameters, and the trip blank will be analyzed only for VOCs. The MS and MSD will be analyzed for all parameters except inorganics, which will have an MS and a lab duplicate. The total number of analyses, including QA samples, for surface water samples is presented in Table 3-12.

Sediment sampling should be concentrated in depositional areas, specifically where finer grained materials exist. Each of the sediment samples will be analyzed for the full suite of analytes listed in Table 3-13. The field duplicate and rinsate blank will be analyzed for all parameters except TOC and geotechnical parameters. The trip blank will be analyzed only for VOCs. The MS and MSD will be analyzed for all parameters, except TOC, metals, and geotechnical parameters. The total number of

laboratory analyses, including QA samples, is presented in Table 3- 14. For volatile fractions only, sediment samples collected from areas with no standing water will be collected and containerized in EnCore™ samplers as noted in Tale 3-15.

3.4.3.1 Mine Fill A Surface Water / Sediment Sampling

A total of 15 surface water / sediment samples (12SW/SD01 through 12SW/SD15) are proposed for this SWMU. A total of nine sediment samples (12SD16 through 12SD24) will be collected to determine the impact, if any, of long-term historical discharges. This SWMU is located at a topographic high point and all surface water that exists at the SWMU originated within the SWMU boundary. The surface water/sediment samples are located in all apparent surface water conveyances at the SWMU along the SWMU boundary to address the majority of surface water runoff. All of these samples are intended to address SWMU related contamination. Therefore, it will be concluded that organic contamination, if present in this medium would be site related. Organic contamination results would be subject to comparison to regional background data sets to determine site related contamination.

3.4.3.2 Mine Fill B Surface Water / Sediment Sampling

The sampling approach at Mine Fill B is similar to the approach taken at Mine Fill A. A total of 13 surface water/sediment samples (13SW/SD01 through 13SW/SD13) are proposed for this SWMU. Background will be evaluated in the same manner as the Mine Fill A. A total of nine sediment samples (13SD14 through 13SD22) will be collected to determine the impact, if any, of long-term historical discharges.

3.4.3.3 Cast High Explosives Fill Surface Water / Sediment Sampling

A total of seven surface water/sediment samples (16SW/SD01 through 16SW/SD07) are proposed for this SWMU in the ditches that exit this SWMU. Two of the sample locations (16SW/SD02 and 16SW/SD07) are located at the discharge point of the sumps located at the SWMU. A total of four sediment samples (16SD08 through 16SD11) will be collected to determine the impact, if any, of the long-term historical discharges. This SWMU is also located at a typographic high and the contamination results will be evaluated in respect to background in the same manner as Mine Fills A and B. All surface water/sediment at the SWMU is derived from within the SWMU boundary, and the collection of SWMU-specific background samples and associated evaluation is not appropriate.

3.4.3.4 Pyrotechnic Test Areas Surface Water / Sediment Sampling

As previously discussed, the Pyrotechnic Test Area lies within and along both sides of the floodplain of Boggs Creek. A total of 29 surface water/sediment samples (19SW/SD01 through 16SW/SD27) are proposed for this SWMU, which includes sampling in ditches which are tributaries to Boggs Creek located in proximity to treatment areas. Upstream samples are also proposed, including samples 19SW/SD01 and 19SW/SD06 for background comparison.

3.5 INVESTIGATION EQUIPMENT AND PROCEDURES

3.5.1 UXO Screening of Selected Drilling Sites and VOC Screening

Because of the prior uses of the areas to be investigated, there is a possibility that unexploded ordnance (UXO) may be encountered during operations at SWMU 19. All activities at SWMU 19 will be conducted consistent with the UXO procedures discussed in the HASP, with indicated variances. The UXO specialist will conduct a surface sweep in all remaining areas of the site that are not used for regular vehicular or pedestrian traffic prior to the commencement of intrusive activities.

SMWU 19 borings will be screened for UXO to a depth of 2 feet and a magnetic gradiometer (down-hole instrument) or similar instrument will be lowered into the hole to clear the borehole. This clearance procedure will continue until the borehole has been cleared to a depth of 10 feet. Advancement may proceed within a 2-foot radius of the clearance boring. All UXO procedures and borehole clearance will be documented in the site UXO logbook.

3.5.2 DPT Sampling for Surface and Subsurface Soil

DPT (e.g., Geoprobe®) will be used to collect surface and subsurface soil samples from the unconsolidated overburden. The procedure for soil sampling using DPT is included in SOP CTO166-04. A new acetate liner will be used for each 2-foot section of soil core. Each removed soil core will be scanned for VOCs. The soil core will be visually inspected and logged by the field geologist, noting the soil texture, grain size (sand, silt or clay), color (and any unusual discoloration), moisture content, and United States Geological Service (USGS) classification. The soil depositional environment will be identified by the field geologist.

It may be possible that the local geology at a particular SWMU may differ from the background areas that were previously identified, sampled, and analyzed to establish representative metals concentrations (as detailed in TtNUS 2001). For example, the lower Pennsylvanian bedrock (and associated residual soil)

may consist locally of thin black shale, carbonaceous shale, and coal beds. These localized beds may contain elevated concentrations of naturally occurring inorganics (e.g., chromium, arsenic). As a result, residual soil in this localized geological environment may have abnormally high inorganic background concentrations. In these instances, it may be appropriate to collect SWMU-specific background soil samples. The site geologist will note any localized geologic environment, similar to that described above, that is unique to a specific SWMU and will notify the TOM to determine whether SWMU-specific background samples are warranted. U.S. EPA Region 5 will be consulted for concurrence in the event that SWMU–specific soil samples are recommended for collection.

After each soil core is retrieved, the photo-ionization detector (PID) will be used to scan the soil core for the presence of VOCs (see SOP CTO 166-6). The soil cores will be logged by a geologist (see SOP CTO166-07). The VOC sample will be collected from the point along the soil core that had the greatest PID reading, using an Encore™ sampler (see SOP CTO 166-8). If no PID readings are above background, then the VOC sample will be collected from any discolored area of the soil or from the midpoint of the core. However, VOC aliquots will be confined to depths of 0.5 to 2.0 feet bgs in the case of surface soil samples. The Encore™ samples will be collected from each core immediately after PID readings are collected. When the sample for VOC analysis has been collected, the remaining aliquot will be placed in a decontaminated stainless-steel mixing bowl; rocks, gravel, and other coarse debris will be removed; and the sample will be mixed with a decontaminated stainless-steel spoon. The appropriate sample jars for remaining analysis will then be filled and properly labeled. The bowl and spoon will be decontaminated between each sample. Details regarding the collection and labeling of soil samples are included in SOP CTO166-1.

Up to two subsurface soil samples will be collected from each soil boring. Soil sample analytical information is presented in Table 3-2, and appropriate containers to be used for each sample aliquot are listed in Table 3-15. When the samples are containerized, labeled, tagged, and bagged (see SOPs CTO166-01 and 03), they will be placed in a cooler containing ice until they can be packaged and prepared for shipment (Section 3.7).

For each cooler containing soil samples identified for VOC analysis (i.e., Encore™ samplers), a trip blank must also be stored in the cooler and must continuously accompany the VOC samples until they have been analyzed. As samples are added to a cooler, the chain-of-custody form will be updated to include each new sample container (per SOP CTO166-03; see also Section 4.0).

One duplicate soil sample will be collected for every 10 soil samples. Soil duplicates will be collected for those samples that have the greatest probability of containing contaminants (e.g., elevated levels of

VOCs as indicated by the PID reading). The duplicate samples should be placed in the same type of containers and handled in the same manner as the regular soil samples. The duplicate samples will be given unique QC sample identifications (see SOP CTO166-02). One rinsate blank will be collected for every 10 soil samples. For soil samples, the rinsate blank will consist of running distilled water over a decontaminated sampling spoon into a decontaminated mixing bowl. This water will be used to fill sample bottles for the rinsate blank. This process will continue until all the necessary bottles are filled for the rinsate sample (see Tables 3-3 and 3-16).

Soil borings will be backfilled with either the soil cuttings (if uncontaminated) or bentonite pellets (if contaminated) in accordance with Section 3.11.

When a boring has been sampled and backfilled, it will be identified by a tall wooden lath driven into the soil near the boring; a 2-by-2-inch inch wooden stake will be driven into the center of the backfilled boring. The stake and the lath will both have brightly colored flagging attached to them to increase visibility, and both will be labeled using a waterproof marker with a unique soil boring number, corresponding to the boring log containing the survey data for the boring.

3.5.3 Inspection and Repair of Existing Monitoring Wells

Existing monitoring wells at SWMU 16 (WES-14-01-83 through WES-14-05-83) will be sampled as part of this investigation. These wells may have broken casings, bent casings, missing caps, no locks, broken locks, obstructions, and other problems. In addition, it is likely that some of the wells may contain enough sediment at the bottom that the well screen may be partially or totally clogged. Therefore, at the beginning of field activities, the existing wells will be located, inspected, and repaired if possible. For example, broken or bent polyvinyl chloride (PVC) riser pipes above the ground surface will be sawed off and new riser pipes will be added. Caps and locks will be replaced if necessary. The height of casing above ground surface and the depth to the bottom of the well will be measured and recorded. This information will be compared to the boring logs and well construction logs to confirm that the wells are open through the screen interval. These activities are addressed in SOP CTO166-09. Each of the existing wells will need to be redeveloped (see SOP CTO166-10) before any sampling can occur. After sampling all existing wells will be resurveyed.

3.5.4 Well Drilling and Installation

A total of 57 shallow wells will be installed at SWMUs 12, 13, and 16, and 30 wells may be installed at SWMU 19 as part of this investigation. Shallow well borings that do not encounter bedrock will be drilled

using DPT or hollow-stem auger drilling with split-spoon soil sampling. In some locations at SWMUs 12, 13, and 16, bedrock may be reached before shallow ground water is encountered. At these locations, monitoring wells will be completed as bedrock installations using rotary drilling.

At SWMUs 12, 13, and 16, where bedrock well installations are anticipated, up to two (SWMU 16) or four (SWMUs 12 and 13) well borings will be continuously sampled using hollow-stem auger drilling with split-spoon soil sampling or DPT through the soil until bedrock is reached; this will be continued with rotary coring to the final depth (minimum 2-inch-diameter samples). The soil samples and bedrock cores should provide an accurate characterization of stratigraphy, fracture distribution, and other features of the soil and rock units spanning the study area. Procedures for drilling and logging a boring in rock are included in SOP CTO166-11. After these holes have been cored, they will be enlarged to a minimum diameter of 5 inches using an air rotary drill rig. Remaining borings that extend into bedrock will be drilled using air rotary methods. Geologic logging of these borings will be based on the rock chips brought up with the circulating air (SOP CTO166-11). In addition, the rates of water produced during drilling will be carefully observed in order to identify rock zones that might be fractured and have above-average permeability characteristics. Drilling standby may be required to monitor for the presence of ground water.

Either a temporary steel casing or hollow-stem auger flights will be placed through the overburden and into the bedrock to minimize collapse and to minimize downward vertical contaminant migration during drilling of the bedrock borings. The temporary casings or auger flights will be removed just after the installation of the permanent PVC riser pipe, sand pack, and bentonite pellet seal; and immediately before installation of the cement-bentonite grout.

At least three each of the shallow well borings at SWMUs 12, 13, and 16 (to be identified by the field geologist) will be sampled continuously through the overburden, using either DPT or split-spoon sampling, until the total depth is reached or bedrock drilling is conducted. The remaining shallow well borings may be sampled at 5-foot intervals or as determined by the field geologist. Bedrock drilling, if required in the shallow well borings, can be conducted using a conventional rotary drilling with a roller bit and water wash.

A permanent monitoring well will be installed in each of the well borings. The wells will be constructed of 2-inch-diameter, Schedule 40, flush joint, PVC riser pipe and slotted screen (see SOP CTO166-12). In each well, the screen will be 10 feet long and have a slot size of 0.02 inch (factory slotted). All riser pipe and screen sections will be new and certified clean. Each well will consist of slotted screen and enough solid riser pipe above so that the riser will extend approximately 2.5 feet above the ground surface.

A sand filter pack will be placed around the annulus of the well screen, from the bottom of the hole upward to 3 feet above the top of the well screen. Three feet of bentonite chips will be placed above the sandpack, and they will be allowed to wet and expand for at least 3 hours before grout is added to the hole. After at least 3 hours has elapsed, the remainder of the well annulus will be filled up to the ground surface with a cement-bentonite grout mixture. A tremie pipe will be used to place grout in the well annulus to help ensure a good seal around the well annulus. An outer, black steel protective casing will be installed around the PVC casing. The outer casing will extend at least 2 feet below the ground surface and no more than 4 inches above the inner PVC well cap. The steel casing will have a hinged cap, or removable cap, and a padlock. A 6-inch-thick, 3-by -3-foot concrete surface pad will be placed around the well at the ground surface. In addition, three barrier posts will be installed around the concrete pad. Details regarding well construction sand pack and grout materials, the outer protective casing, the well pad, and the protective barrier posts are included in SOP CTO166-12.

3.5.5 Packer Testing of Uncased Borings

At SWMUs 12, 13, or 16, where bedrock well installations are anticipated, the cored well borings may also be packer tested. The decision to perform packer testing will be based on information collected during drilling (fractures, water yield, and/or total footage cored) that would warrant a more detailed evaluation of specific water-yielding zones in the bedrock. A double inflatable packer assembly attached to the drill string of the drilling rig will be used to hydraulically test specific sections of each new uncased boring. The procedures for conducting a packer test are presented in SOP CTO166-13. The packer assembly will be thoroughly decontaminated before being placed in a boring. The inflatable packers will be set 5 to 10 feet apart (i.e., the vertical section of rock tested in a single test is 5 to 10 feet). The packer assembly will be lowered down the boring so that the packers isolate a section of the hole that was identified during drilling as being fractured or yielding above-average rates of ground water. Two to three different zones will be tested for each hole, as determined by the field geologist. The number and depths of the zones to be tested will be determined by the FOL and lead field geologist based on their best judgment.

The packer tests will be the pump-out type, where ground water is pumped out of the hole from the isolated section of boring. A pump positioned between the two packers will pump water out of the packed-off interval. Thus, no external water will be introduced to the holes during the packer tests. The flow rate of water discharging from the pump and the hydraulic pressure within the pack-off interval will be measured frequently during the test. The pumping rate will be held nearly constant. Initially, the pumping rate will be at a low level (e.g., 1 gallon per minute or less). If this pumping rate can be sustained for 15 minutes and the water level stabilizes, then the rate will be increased to about 5 gallons per minute.

This rate will be maintained and the hydraulic head will be monitored for another 15 minutes. After 15 minutes, the pumping rate can be increased if the aquifer can sustain the increased rate and the FOL or lead geologist determines that a higher rate is necessary.

The data will be used to determine a hydraulic conductivity value for the rock material. They will also be used to help choose a suitable interval in the hole for the installation of the monitoring well screen.

3.5.6 Well Development

All existing and any new monitoring wells will be developed in accordance with SOP CTO166-10 to remove fine sediment from inside and around the well screens. The method used to perform well development can be either vigorous on-and-off pumping or surge block and pumping, depending on which technique is most effective. Field measurements of pH, specific conductance, temperature, and turbidity will be collected at 5- to 10-minute intervals on the water retrieved from the well, in accordance with SOP CTO166-14. Well development will continue until the well water is clear to the unaided eye, and following this three subsequent consecutive readings of pH, specific conductance, and temperature are within 10 percent of each other and three consecutive turbidity readings are within 5 NTUs of each other. If the water quality parameter criteria cannot be met after five well volumes of water have been removed, then one additional well volume will be removed and well development will be considered complete.

All water removed from the well during the development process will be stored in a portable holding tank (already present at NSWC Crane) and discharged into a designated man-hole (see SOP CTO166-15).

3.5.7 Water-Quality Field Measurements

Field measurements of water-quality parameters, including pH, specific conductance, ORP, temperature, DO, and turbidity, and nitrate, will be collected from ground water in a flow-through cell attached to a pump discharge line. These measurements will be performed during well development (Section 3.5.6) and low-flow well sampling (Section 3.5.8). These field measurements will be made using a YSI 6-Series Environmental Monitoring System or equivalent type of instrument. Turbidity measurements will be made using a LaMotte turbidity meter or equivalent. Calibration and measurements made with the field instruments will be in accordance with Section 6.1 and SOP CTO166-14.

Each calibration of an instrument will be recorded on an equipment calibration log sheet. Water-quality measurements, along with date, time, instrument operator, and visual and other observations (e.g.,

weather conditions) will be recorded on well development logs, sample collection logs, or in field notebooks, as appropriate.

3.5.8 Low-Flow Sampling of Monitoring Wells

Low-flow sampling procedures will be used to collect ground water samples from existing wells and new wells (see SOP CTO166-05 and -16). If the depth to ground water is greater than 20 feet, a compressed air bladder pump or submersible pump will be used to slowly pump ground water from the well at about 100 milliliters per minute (mL/minute). Low-flow pumping will proceed until readings of water temperature, pH, specific conductance, dissolved oxygen, ORP, and turbidity stabilize but for no longer than 4 hours. Appropriate sample bottles will be filled directly from the pump discharge tube (see Tables 3-4 and 3-16). The bottles will be properly labeled, tagged, bagged, and placed in an ice-filled cooler as quickly as possible (see SOPs CTO166-01 and CTO166-03). A ground water collection log sheet will be completed for each ground water sample collected (see SOP CTO166-05).

The wells will be sampled in order of least contaminated to most contaminated, starting with the upgradient wells, proceeding to the far downgradient wells, and finishing with the source wells. This approach will lessen the possibility of incurring cross contamination between wells. After each ground water sample is collected, the pump will be decontaminated (SOP CTO 166-17). For every 10 ground water samples that are collected, a rinsate blank sample will be collected to ensure that decontamination of the pumps is effectively accomplished. The rinsate blank will be collected by pumping distilled water through the pump and placing the pump discharge water directly into sample jars. The appropriate sample jars and preservatives for each analyte group are listed in Table 3-16.

For each cooler containing samples identified for VOC analysis, a trip blank will be included in the cooler. The trip blank will accompany the VOC samples until they have been analyzed for VOCs. As samples are added to a cooler, the chain-of-custody form should be updated to include each new sample container (see SOP CTO166-03).

3.5.9 Ground-Water-Level Measurements

Two synoptic rounds of water-level measurements and total well depth soundings will be obtained at each of the new and existing monitoring wells in the vicinity of each SWMU. All water-level measurements will be taken within a eight-hour period using an electronic water-level meter. Water-level elevations will be recorded to within a 0.01-foot accuracy from a marked reference point on the well riser pipe. Detailed procedures regarding water-level measurements are included in SOP CTO166-18. Water levels will be

recorded on a TtNUS water-level form. A blank water-level form is provided in SOP CTO166-18 for reference purposes. The water-level meter will be decontaminated between each well; decontamination procedures are addressed in SOP CTO166-17.

Two rounds of water level measurements will be collected in all wells and staff gages at SWMU 19 (if installed). One round will be collected during dry conditions and the other round will be collected immediately after a precipitation event.

3.5.10 Surface Water Sampling

A total of 60 surface water samples will be collected as grab samples from SWMUs 12, 13, 16, and 19 in accordance with SOP CTO166-19. It will be assumed that the water flowing at the sample locations is thoroughly mixed. Water sample bottles will be filled directly in the flowing water. If the depth of water is not sufficient to lower the bottles into the water without stirring up bottom sediment, then a decontaminated shovel will be used to dig a depression (i.e., a small pool) about 2 feet deep where the water can accumulate and pass through. After suspended sediment in the pool has settled out or is carried downstream, the unfiltered surface water sample will be collected. A filtered water sample also will be collected for dissolved metals analysis by drawing water into a plastic syringe and forcing it through a 0.45-µm filter cartridge directly into a sample container (see SOP CTO166-19). The bottles will be properly labeled (see SOP CTO166-01), tagged, bagged, and placed in an ice-filled cooler.

Field water-quality measurements (see Section 3.5.7) will be made at each sampling location immediately after a sample has been collected. These data will be recorded on the sample collection log sheet.

3.5.11 Sediment Sampling

A total of 60 sediment samples will be collected as grab samples from SWMUs 12, 13, 16, and 19 in accordance with SOP CTO166-20. If no water is flowing at a sampling location, the sample may be collected where fine sediment has accumulated. If water is flowing at the sampling location, then the sample will be collected at the edge of the flowing water along depositional areas, where fine sediment has accumulated. The appropriate sample jars will be filled directly from the sediment surface using disposable plastic spoons. Sample materials will be collected from 0 to 4 inches beneath the ground surface.

As samples are added to a cooler, the chain-of-custody form will be updated to include each new sample container (see SOP CTO166-03). A sample collection log sheet will be completed for each sediment

sample collected. These logs will note the date, time, sampling personnel, weather conditions, and flow conditions at the sampling location and will include a complete description of the sampling location (see SOP CTO166-20). A 2- by 2-inch wooden stake with attached flagging material will be driven into the sampling location, and the sample identification number will be marked on the stake with a black waterproof marker. Alternatively, stakes may be driven into the bank to mark the location of the sediment sample. This will be done so that the sampling location can be revisited at a later date, if necessary, for surveying or resampling purposes.

3.5.12 Staff Gage Installation and Estimation of Stream Flow

At least six staff gages will be installed at each of the SWMUs. The staff gages will be placed approximately equidistant along the stretch of each significant water body containing water for adequate spatial distribution. Existing permanent structures, such as culverts and bridge abutments, where measurements to the water surface can be taken will be used as staff gages as much as possible. A mark will be placed on these permanent structures denoting the point where measurements will be taken and the staff gage number will be noted. If a permanent structure is not available, the staff gage will consist of a metal pipe that will be pounded into the sediment with a sledgehammer. The measurements will be taken with a weighted tape measure in reference to the established measuring point. All staff gages will be measured at the same time as water-levels in monitoring wells. Staff gages and monitoring well water levels will be measured in two rounds, one during dry conditions and one immediately after a precipitation event.

The approximate flow volume of surface water at each staff gage location will be estimated during both rounds by measuring the cross section of the streambed and measuring the surface water flow, in accordance with SOP CTO 166-21. The information will be used to evaluate surface water flow in relation to the sites.

3.5.13 Aquifer Testing

Aquifer testing, in the form of slug tests, will be performed in up to six of the shallow wells in each of SWMUs 12, 13, and 19 and in up to three shallow wells at SWMU 16. Both rising and falling-head tests will be performed in wells with submerged well screens, whereas only rising-head tests will be performed in wells that straddle the water table. The slug tests will be performed in accordance with SOP CTO 166-22.

3.6 QUALITY CONTROL SAMPLES

This section focuses on field QC samples that will be collected as part of this environmental investigation. Field QC samples include field duplicates, source water blanks, equipment rinsate blanks, and trip blanks. Tables 3-3, 3-5, 3-12, and 3-14 present the types and numbers of required field QC samples to be collected for soil, ground water, surface water, and sediment sampling activities, respectively, during the field investigation. Table 3-17 provides an overview of QA frequencies and corrective action measures. Section 8.0 provides definitions and details for these and all other QC checks to be used during this investigation. Field QC sample requirements for field duplicates, source water blanks, equipment rinsate blanks, and trip blanks required for this project are described below.

Field Duplicates. Field duplicates are obtained during a single act of sampling and are used to assess the overall precision of the sampling and analysis program. Duplicate samples will be collected at a rate of one for every 10 environmental samples of each type of environmental medium. All duplicate samples will be analyzed for the same parameters in the laboratory as their environmental sample counterparts. Duplicate samples will be preferentially collected where field evidence (i.e., PID reading or odor) indicates that contamination is likely to be present in the environmental sample.

Equipment Rinsate Blanks. Equipment rinsate blanks will be obtained under representative field conditions by running analyte-free water through sample collection equipment after decontamination and placing it in the appropriate sample containers for analysis. Equipment rinsate blanks will be collected for non-dedicated equipment for all sampling rounds. For surface and subsurface soil sampling activities, rinsate blanks will be collected by running analyte-free water over a decontaminated stainless-steel bowl and mixing spoon; these items are used to mix soil before it is placed in sample jars. For ground water samples, analyte-free water will be pumped through a decontaminated pump and tubing that will be used for ground water sampling. Equipment rinsate blanks will not be required for surface water and sediment samples. Surface water samples will be collected directly from the surface water body, and only a new disposable spoon will be used to collect sediment samples.

Source Water Blanks. Source water blanks will be obtained by sampling each water source (e.g., potable water and distilled water) used for decontamination activities during the field investigation. Source water blanks will be used to determine if the water or the laboratory bottles are contributing to sample contamination. Source water blanks will be collected for each type of water used for decontamination and will be submitted at a frequency of one per sampling event. Source water blanks, as applicable, will be analyzed for the entire suite of parameters under investigation. It is anticipated that two source water

blanks will be collected during the field investigation: one potable water sample and one sample of distilled water used for decontamination.

Trip Blanks. Trip blank samples are 40-mL glass vials that contain analyte-free water and are prepared by the analytical laboratory prior to the start of field activities. They should be stored in a sealed container until they are needed. During sampling activities, one trip blank, consisting of one vial, will be placed in each cooler that contains environmental samples destined for VOC analysis. The trip blank will be properly labeled and added to the chain-of-custody form belonging to the cooler. Trip blanks are only analyzed for VOCs.

3.7 SAMPLE HANDLING, PACKAGING, AND SHIPPING

3.7.1 Sample Preservation

Preservation requirements for each of the chemicals of interest are provided in Tables 3-15 and 3-16. All soil and sediment samples require only to be cooled to 4 ± 2°C; no chemical preservatives are necessary. Sample bottles for aqueous samples will contain the proper amounts and types of preservatives before they are shipped to NSWC Crane (Table 3-16). The preservatives placed in the sample bottles will be certified that they are free of analytes being tested in the samples. All samples will be promptly chilled with ice to 4°C ± 2°C and packaged in an insulated cooler. Each cooler will include a temperature blank. Ice will be sealed in containers to prevent water leakage. Samples will not be frozen.

3.7.2 Sample Labeling

Before samples are packaged, the sample labels and tags will be checked to ensure that all information on the label and tag is complete and correct (see SOP CTO166-01). This information will be checked to ensure that it matches the information placed on the sample collection log sheets and the chain-of-custody form.

3.7.3 Sample Packaging

Each sample container will be placed in a zip-lock bag to prevent cross-contamination or leakage. The zip-lock bag will be placed in a bubble-wrap sleeve to protect from breakage and cross-contamination. Only shipping containers that meet all applicable state and federal standards for safe shipment will be used. Cube ice will be placed in plastic bags and placed around and between the samples in sufficient quantity to ensure that the samples remain chilled (4°C ± 2°C) during transport to the analytical laboratory.

The completed field chain-of-custody document will be signed, placed in a sealed plastic envelope, and taped to the top inside cover of the shipping container (see SOP CTO166-03).

SOP CTO166-01 provides a detailed description of sample handling, packaging, and shipping procedures required for this project. The FOL will be responsible for ensuring the completion of the following forms:

Sample Labels and Tags
Chain-of-custody Forms
Custody Seals for Coolers
Shipping Labels for Coolers
Express Mail Air Bills

3.7.4 Sample Shipping

Shipping containers (i.e., coolers) will be sealed with nylon strapping tape in at least two places, and custody seals will be signed, dated, and affixed in a manner that will allow the receiver to quickly identify any tampering that may have occurred during transport to the laboratory (see SOPs CTO166-01 and -03).

Shipment will be made by a public courier at the next scheduled pickup following completion of sample collection. Copies of the Express Mail Air Bills should be retained by the FOL for tracking purposes, if needed, and for communications with the laboratory. Air Bills will be retained for the Permanent Record File.

3.7.5 Sample Custody

Custody of samples must be maintained and documented at all times in accordance with SOP CTO166-03, beginning with the collection of samples in the field. Sample custody procedures are addressed in Section 4.0.

3.8 RECORD KEEPING

Standard forms, field notebooks, and a field logbook will be used to record all sample collection activities, field measurements, observations concerning site conditions, and other project-related information. These records include sample log sheets, daily activity records, field logbooks, drilling and well completion log sheets, and field instrument calibration log sheets, among others. More details regarding record keeping are included in SOP CTO166-03.

3.8.1 Field Log Books

Bound, weatherproof field notebooks will be maintained by sampling personnel. All information related to sampling and other field activities will be recorded in field notebooks. This information will include, but is not limited to, sampling time, weather conditions, unusual events, field measurements, and descriptions of photographs.

A bound, weatherproof logbook will be maintained by the FOL. This book will contain a summary of each day's activities and will reference the field notebooks when applicable.

3.8.2 Drilling and Well Completion Logs

A drilling log will be completed for every boring that occurs during these field activities. A geologist will complete the boring log, which will include information regarding date, time, personnel, drilling and sampling equipment, geologic materials encountered, fracture locations and density in bedrock (where appropriate), color, texture, odors, and readings made with the screening instruments (see SOPs CTO166-07 and CTO166-11).

A well completion log will be completed for every monitoring well that is constructed. These logs will include information concerning the date, time of events, quantities of construction materials used, lengths and diameters of riser pipe and well screen placed in the well, and other information, as described in SOP CTO166-12.

3.8.3 Well Development Log Sheets

During the development or redevelopment of each monitoring well, the date, time of events, development method and equipment, personnel present, amounts of water produced, measurements made by field water-quality meters, and depths to water will be recorded on a well development log sheet, as described in SOP CTO166-10.

3.8.4 Equipment Calibration Logs

An equipment calibration log sheet will be used to record each time an instrument is calibrated or recalibrated or calibration is checked against a standard or background. The procedures and standards to be used for instrument calibration are discussed in Section 5.1 and each instrument's SOP is contained in Appendix H.

3.8.5 Sample Collection Logs

One sample collection log sheet will be completed for every environmental sample, every duplicate sample, and every field blank sample collected during the field activities. Only the MS and MSD samples do not require their own individual sample collection log sheet.

3.8.6 Chain-of-Custody Forms

A chain-of-custody form will be completed for every cooler that contains samples being shipped to an off-site laboratory for analyses. These forms are a record of the people having custody of the samples from the time the samples are collected to the time they are analyzed and disposed (see SOP CTO166-03). The completed field chain-of-custody document will be signed, placed in a sealed plastic envelope, and taped to the top inside cover of the shipping container before it is shipped. A copy of the document will be retained by the FOL.

3.8.7 Shipping Forms/Air Bills

Copies of all forms and/or Air Bills related to the shipment of coolers will be retained by the FOL in order to trace the shipment, if necessary, and to communicate with the receiving laboratory.

3.8.8 Permanent Record File

At the completion of the field activities, the FOL will submit to the TOM all field records, data, field notebooks, logbooks, COC records, sample log sheets, daily activity logs, and other records concerning the project, including all the forms and log sheets listed above. The FOL will check these records for legibility and completeness before submitting them to the TOM. These forms, data, and field notes will become part of the permanent project record.

3.9 SURVEYING

The location of every soil boring, surface water, and sediment sample will be marked with a wooden lath and flagging, and a hole number will be marked on the lath. In addition, a 2- by 2-inch wooden stake, 6 inches long, will be driven into the ground at the center of the backfilled boring. This stake will have a piece of brightly colored flagging tacked onto its top, and the hole number will be indelibly marked on the side of the stake.

The horizontal and vertical locations of all monitoring wells both pre-existing and new, soil borings, surface water samples, sediment samples, and staff gages will be surveyed. The horizontal location will be surveyed for all locations to the Indiana State Plane Coordinates within the nearest foot and referenced to the 1983 North American Datum (NAD83). The vertical elevations of the ground surface and top of casing for wells, the top of the staff gage, and the ground surface for the borings and surface water and sediment samples will be measured to the nearest 0.01 foot.

3.10 EQUIPMENT DECONTAMINATION

All equipment used to collect soil and ground water samples will be decontaminated in accordance with SOP CTO166-17.

3.11 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

It is anticipated that this investigation will generate five types of potentially contaminated residues or investigation-derived waste (IDW):

- Personal protective equipment (PPE)
- Ground water sample tubing and DPT sample liners
- Well development and purge fluids
- Equipment decontamination fluids
- Waste cuttings from drilling activities

IDW will be handled as described below:

PPE, Tubing, and DPT Sample Liners - All PPE, tubing, and DPT sample liners will be decontaminated and double bagged and placed in trash receptacles at the facility.

Well Development and Purge Fluids - All well development and purge fluids will be collected, stored, and discharged to the NSWC Crane permitted sanitary sewer system in accordance with SOP CTO166-15.

Drilling Equipment Decontamination Fluids - All drilling equipment decontamination fluids will be combined with well development and purge fluids and handled in the same manner as described for well development and purge fluids.

Equipment Decontamination Fluids - All sampling decontamination fluids will be combined with well development and purge fluids and handled in the same manner as described for well development and purge fluids.

Waste Cuttings and Rock Cores from Drilling Activities - For each boring, the obtained cuttings will be scanned for VOCs. If all readings from these screening instruments are at background levels, the soil cuttings from soil borings will be mixed with bentonite, placed back down the hole, and tamped for compaction or spread on the ground adjacent to the borehole. Borings that contain contaminated cuttings will be backfilled with bentonite pellets or a cement-bentonite slurry.

Contaminated cuttings from borings will be placed in black plastic trash bags (or directly in drums if larger quantities), labeled, and then placed in 55-gallon sealable drums. The bags will be tagged and the drums will be clearly labeled regarding the dates, locations, and depths from where the soils originated and the personnel (including phone number) placing the cuttings in the drum. When analytical results concerning contaminant concentrations in the soil samples are received back from the laboratory, decisions will be made as to how to dispose of the soil materials (see SOP CTO166-15).

3.12 SAFETY

Health and safety issues and concerns are critically important during any field investigation involving drilling, UXO, and hazardous wastes. Sampling team members working at the site must be fully aware of the potential dangers involved with sampling activities, must be trained and prepared to deal with problems or health-related issues as they arise, and should minimize to the greatest possible extent the potential for exposure to harmful chemicals or accidents. To ensure that field activities are performed at a high level of safety, the following are included in the health and safety activities related to the sampling program.

3.12.1 Health and Safety Plan

A separate HASP has been prepared that describes specific health and safety requirements, concerns, and information related to the site activities. This document will be read and understood by each person working at the site. Each worker or visitor to the site must sign an acknowledgment that he or she has read and understands the HASP.

3.12.2 Health and Safety Training

All workers involved with the site investigations shall have successfully completed the OSHA-mandated 40-hour health and safety training and the follow-up annual 8-hour refresher courses when appropriate.

TtNUS and subcontractor personnel will supply OSHA documentation before beginning work. Personnel who do not comply with this requirement must receive verbal approval to work from TtNUS corporate health and safety personnel.

3.12.3 Personal Protective Clothing and Equipment

Workers at the site must be part of a medical monitoring program and must be medically approved to perform their duties without physical limitations. Protective clothing and equipment, as specified in the HASP, will be worn while performing site activities.

3.12.4 Safety Meetings

Safety meetings will be held among on-site workers whenever the SSO feels it is appropriate. The SSO will discuss safety issues related to activities being performed and will make site workers aware of any new conditions that could potentially affect health or safety.

3.13 ORGANIZATION AND LOGISTICS OF FIELD INVESTIGATIONS

3.13.1 Personnel

The duties, responsibilities, and line of command for each person working on the project are described in Section 2.0 and displayed on Figure 2-1. Persons working on the project will be intimately familiar with their roles and responsibilities. In addition, they will be familiar with the mechanisms and procedures for coordinating tasks, improving communications, and reporting incidences or irregularities. The FOL is responsible for coordinating all on-site personnel and activities (Section 2.3.1). The SSO is responsible for health and safety monitoring and ensures that the HASP is adhered to during all field activities (Section 2.3.3). The SSO has the authority to stop work if an imminent safety hazard is encountered (Section 2.3).

3.13.2 Subcontractors

Subcontractors will perform site activities involving drilling, packer testing, well installation, and surveying. The FOL will direct all subcontractor activities.

3.13.3 Mobilization and Demobilization

Following approval of the QAPP, TtNUS will begin mobilization activities. All field team members will review the QAPP (including the HASP) prior to mobilization. In addition, a field team orientation meeting will be held to familiarize personnel with the scope of the field activities. Items to be presented during that meeting include:

- Identification of the QAPP, including the HASP and applicable field SOPs (Appendix H).
- Site-specific safety concerns and requirements.
- Project objectives.
- Sampling designs and strategies for soil and ground water (including the relationship of soils data for this project to NSWC Crane background soil data).
- Site-specific particulars of field operations (e.g., locations of utilities, physical access to sampling location, communication mechanisms, lines of authority and responsibility, scheduling requirements, sample shipping concerns, etc.).
- Laboratory and other subcontractor coordination.
- Site access requirements.
- Travel requirements.

The FOL will coordinate the mobilization activities for this project. The equipment required for the field activities will be mobilized from the TtNUS Pittsburgh office or a third-party vendor. The FOL and crew will demobilize from the site upon completion of the field operations and transport field equipment back to the TtNUS Pittsburgh office, as necessary. All areas will be thoroughly checked and trash will be removed and disposed properly.

TABLE 3-1

FIELD SCREENING AND MEASUREMENTS
SWMUs 12, 13, 16, AND 19
NSWC CRANE, CRANE, INDIANA

Parameter	Equipment	SOP	Calibration
Volatile Organics	Perkin-Elmer Photovac 2020 Photoionization Detector	SOP CTO166-6	Per Manufacturer
pH, Temperature, oxidation-reduction potential, specific conductance, dissolved oxygen (water quality parameters)(1)	YSI 6-Series Environmental Monitoring System	SOP CTO166-14	Per Manufacturer
Turbidity (water quality parameter)(1)	LaMotte Turbidity Meter	SOP CTO166-14	Per Manufacturer
Water-Level Measurements	Heron Dipper-T (or equivalent)(2)	SOP CTO166-18	Per Manufacturer
Unexploded Ordnance (Surface) (3)	GA-72CV Magnetic Locator	Per Manufacturer	Per Manufacturer
Unexploded Ordnance (Subsurface) (3)	MG-220 Magnetic Gradiometer	Per Manufacturer	Per Manufacturer

1 Field measurements used to establish well stabilization prior to collecting ground water samples. Field measurements will also be collected at each surface water sample location.
2 Any electronic water-level indicator with the capability of measuring to depths of 100 feet may be used.
3 SWMU 19 only.

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
SWMU 12																			
12SB01	0-2 ft bgs	1	1	1	1	1	1					1	1	1					
	above water table	1	1	1	1	1	1					1	1	1					
12SB02	0-2 ft bgs															1	1	1	1
	above water table															1	1	1	1
12SB03	0-2 ft bgs															1	1	1	1
	above water table															1	1	1	1
12SB04	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB05	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB06	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB07	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB08	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB09	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB10	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
12SB11	0-2 ft bgs						1					1	1						
	1st above water table						1					1	1						
	2nd above water table						1					1	1						
Total Samples SWMU 12		2	2	2	2	2	26	0	0	0	0	26	26	2	0	4	4	4	4
SWMU 13																			
13SB01	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB02	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
13SB03	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB04	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB05	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB06	0-2 ft bgs	1	1	1	1	1			1		1								
	0-4 ft bgs	1	1	1	1	1			1		1								
13SB07	0-2 ft bgs															1	1	1	1
	above water table															1	1	1	1
13SB08	0-2 ft bgs															1	1	1	1
	above water table															1	1	1	1
13SB09	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB10	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB11	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB12	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB13	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB14	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB15	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
13SB16	0-2 ft bgs			1	1	1			1		1								
	0-4 ft bgs			1	1	1			1		1								
Total Samples SWMU 13		2	2	28	28	28	0	0	28	0	28	0	0	0	0	4	4	4	4
SWMU 16																			
16SB01	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1						
	TBD	1		1	1	1	1	1			1	1	1						
16SB02	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1			1	1	1	
	TBD	1		1	1	1	1	1			1	1	1			1	1	1	
16SB03	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1						
	TBD	1		1	1	1	1	1			1	1	1						
16SB04	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1						

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 3 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
	TBD	1		1	1	1	1	1			1	1	1						
16SB05	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB06	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB07	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1			1	1	1	
	TBD			1	1	1	1	1				1	1			1	1	1	
16SB08	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB09	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB10	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB11	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB12	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1			1	1	1	
	TBD			1	1	1	1	1				1	1			1	1	1	
16SB13	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB14	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB15	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB16	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB17	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB18	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1			1	1	1	
	TBD			1	1	1	1	1				1	1			1	1	1	
16SB19	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB20	0-2 ft bgs	1	1	1	1	1	1	1	1			1	1						
	TBD			1	1	1	1	1				1	1						
16SB21	0-2 ft bgs	1	1	1	1	1	1	1	1			1	1						
	TBD			1	1	1	1	1				1	1						
16SB22	0-2 ft bgs	1	1	1	1	1	1	1	1			1	1						
	TBD			1	1	1	1	1				1	1						

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 4 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
16SB23	0-2 ft bgs	1	1	1	1	1	1	1	1			1	1						
	TBD			1	1	1	1	1				1	1						
16SB24	0-2 ft bgs	1	1	1	1	1	1	1	1			1	1			1	1	1	
	TBD			1	1	1	1	1				1	1			1	1	1	
16SB25	0-2 ft bgs	1	1	1	1	1	1	1	1			1	1						
	TBD			1	1	1	1	1				1	1						
16SB26	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB27	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB28	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB29	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB30	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB31	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1			1	1	1	
	TBD			1	1	1	1	1				1	1			1	1	1	
16SB32	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB33	0-2 ft bgs	1	1	1	1	1	1	1	1		1	1	1						
	TBD			1	1	1	1	1				1	1						
16SB34	0-2 ft bgs	1	1	1	1	1	1	1				1	1						
	TBD			1	1	1	1	1				1	1						
16SB35	0-2 ft bgs	1	1	1	1	1	1	1				1	1						
	TBD	1		1	1	1	1	1				1	1						
16SB36	0-2 ft bgs	1	1	1	1	1	1	1				1	1						
	TBD	1		1	1	1	1	1				1	1						
16SB37	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1			1	1	1	
	TBD	1		1	1	1	1	1			1	1	1			1	1	1	
16SB38	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1						
	TBD	1		1	1	1	1	1			1	1	1						
16SB39	0-2 ft bgs	1	1	1	1	1	1	1			1	1	1						
	TBD	1		1	1	1	1	1			1	1	1						
16SB40	1st above water table	1	1	1	1	1	1	1				1	1						
	2nd above water table	1	1	1	1	1	1	1				1	1						

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 5 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
16SB41	1st above water table	1	1	1	1	1	1	1				1	1			1	1	1	
	2nd above water table	1	1	1	1	1	1	1				1	1			1	1	1	
Total Samples SWMU 16		52	43	82	82	82	82	82	28	0	37	82	82	0	0	16	16	16	0
SWMU 19 [3]																			
OTA																			
19SS01	0-2 ft bgs						1					1	1						
19SS02	0-2 ft bgs						1					1	1			1	1	1	
19SS03	0-2 ft bgs						1					1	1						
19SS04	0-2 ft bgs						1					1	1						
19SS05	0-2 ft bgs						1					1	1						
19SS06	0-2 ft bgs	1	1	1	1	1	1					1	1	1					
19SS07	0-2 ft bgs	1	1	1	1	1	1					1	1	1					
19SS08	0-2 ft bgs	1	1	1	1	1	1					1	1	1					
19SS09	0-2 ft bgs	1	1	1	1	1	1					1	1	1		1	1	1	
19SS10	0-2 ft bgs						1			1		1	1						
19SS11	0-2 ft bgs						1			1		1	1						
19SS12	0-2 ft bgs						1			1		1	1						
19SS13	0-2 ft bgs						1					1	1						
19SS14	0-2 ft bgs						1					1	1						
19SS15	0-2 ft bgs						1					1	1						
19SS16	0-2 ft bgs						1					1	1			1	1	1	
19SS17	0-2 ft bgs						1					1	1						
19SS18	0-2 ft bgs						1					1	1						
19SS19	0-2 ft bgs						1					1	1						
19SS20	0-2 ft bgs						1			1		1	1						
19SS21	0-2 ft bgs						1			1		1	1						
19SS22	0-2 ft bgs						1					1	1						
19SS23	0-2 ft bgs						1					1	1			1	1	1	
19SS24	0-2 ft bgs						1					1	1						
19SS25	0-2 ft bgs						1					1	1						
19SS26	0-2 ft bgs						1					1	1			1	1	1	
19SS27	0-2 ft bgs						1					1	1						
19SS28	0-2 ft bgs						1					1	1						
19SS29	0-2 ft bgs						1					1	1						
19SS30	0-2 ft bgs						1					1	1			1	1	1	
19SS31	0-2 ft bgs						1					1	1						
19SS32	0-2 ft bgs						1					1	1						

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 6 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
19SS33	0-2 ft bgs						1					1	1						
19SS34	0-2 ft bgs						1					1	1						
19SS35	0-2 ft bgs						1			1		1	1						
19SS36	0-2 ft bgs						1			1		1	1						
19SS37	0-2 ft bgs						1			1		1	1			1	1	1	
19SS38	0-2 ft bgs						1			1		1	1						
19SS39	0-2 ft bgs						1			1		1	1						
19SS40	0-2 ft bgs						1					1	1			1	1	1	
19SS41	0-2 ft bgs						1					1	1						
19SS42	0-2 ft bgs						1					1	1						
19SS43	0-2 ft bgs						1					1	1						
19SS44	0-2 ft bgs						1					1	1			1	1	1	
19SS45	0-2 ft bgs						1					1	1						
19SS50	0-2 ft bgs						1					1	1						
19SS51	0-2 ft bgs						1					1	1						
19SS52	0-2 ft bgs						1					1	1						
19SS53	0-2 ft bgs						1					1	1						
19SS54	0-2 ft bgs						1					1	1						
19SS55	0-2 ft bgs						1					1	1			1	1	1	
19SS56	0-2 ft bgs						1					1	1						
19SS57	0-2 ft bgs						1					1	1						
19SB01	0-2 ft bgs	1	1	1	1	1	1					1	1	1		1	1	1	
	2-4 ft bgs	1	1	1	1	1	1					1	1	1					
19SB02	0-2 ft bgs						1					1	1						
	TBD						1					1	1						
RR							1					1	1			1	1	1	
19SS58	0-2 ft bgs						1					1	1		1				
19SS59	0-2 ft bgs						1					1	1		1				
19SS60	0-2 ft bgs						1					1	1		1				
19SS61	0-2 ft bgs						1					1	1		1				
19SS62	0-2 ft bgs						1					1	1		1				
19SS63	0-2 ft bgs						1					1	1		1				
19SS64	0-2 ft bgs						1					1	1		1	1	1	1	
19SS65	0-2 ft bgs						1					1	1		1				
19SS66	0-2 ft bgs						1					1	1		1				
19SB03	0-2 ft bgs						1					1	1		1	1	1	1	
	2-4 ft bgs						1					1	1		1				

TABLE 3-2

SUMMARY OF SOIL SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 7 OF 7

	Parameters [1]	Volatiles [2]		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
19SB04	0-2 ft bgs						1					1	1		1				
	2-4 ft bgs						1					1	1		1				
19SB05	0-2 ft bgs						1					1	1		1	1	1	1	
	2-4 ft bgs						1					1	1		1				
19SB06	0-2 ft bgs						1					1	1		1				
	2-4 ft bgs						1					1	1		1				
SWMU 19																			
Annex																			
19SS46	0-2 ft bgs						1					1	1						
19SS47	0-2 ft bgs						1					1	1			1	1	1	
19SS48	0-2 ft bgs						1					1	1						
19SS49	0-2 ft bgs						1					1	1						
19SS67	0-2 ft bgs						1					1	1						
19SS68	0-2 ft bgs						1					1	1						
19SS69	0-2 ft bgs						1					1	1						
19SS70	0-2 ft bgs						1					1	1						
19SS71	0-2 ft bgs						1					1	1			1	1	1	
19SB07	0-2 ft bgs						1					1	1		1				
	TBD						1					1	1		1				
Total Samples SWMU 19		6	6	6	6	6	86	0	0	10	0	86	86	6	19	17	17	17	0

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.
2 Volatile samples will be collected from the 1-2 ft bgs section of all 0-2 ft bgs sampling locations designated for volatile analysis.
3 Up to 120 additional surface soil samples and up to 60 soil borings may be installed and subsurface samples collected at the Pyrotechnic Test Area during Round 2 based on results of Round 1 sampling.

TABLE 3-3

SUMMARY OF SOIL ANALYSES AND QUALITY CONTROL SAMPLES
CTO 166
NSWC CRANE, CRANE, INDIANA

Parameters [1]	Volatiles		Semivolatiles			Explosives		Dioxin	Dyes	PCBs	Metals		Miscellaneous Parameters					
Sample Type	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	SW-846 8290	NSWC Crane	SW-846 8082	Metals SW-846 6020	Mercury SW-846 7471A	Cyanide	Perchlorate	TOC	CEC	pH	Ammonia
Samples SWMU 12	2	2	2	2	2	26	0	0	0	0	26	26	2	0	4	4	4	4
Samples SWMU 13	2	2	28	28	28	0	0	28	0	28	0	0	0	0	4	4	4	4
Samples SWMU 16	52	43	82	82	82	82	82	28	0	37	82	82	0	0	16	16	16	0
Samples SWMU 19	6	6	6	6	6	86	0	0	10	0	86	86	6	19	17	17	17	0
Total Samples All SWMUs	**62**	**53**	**118**	**118**	**118**	**194**	**82**	**56**	**10**	**65**	**194**	**194**	**8**	**19**	**41**	**41**	**41**	8
Field Duplicates [2]	7	6	12	12	12	20	9	6	1	7	20	20	1	2	5	5	5	1
Trip Blanks [3]	10	10	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Rinsate Blanks [4]	7	6	12	12	12	20	9	6	1	7	20	20	1	2	5	5	5	1
Ambient Condition Blanks [5]	1	1	1	1	1	1	1	1	1	1	1	1	1	1	NA	NA	NA	1
Matrix Spikes [6]	4	3	6	6	6	10	5	3	1	4	10	10	1	1	3	NA	NA	1
Matrix Spike Duplicates [7]	4	3	6	6	6	10	5	3	1	4	NA	NA	NA	NA	NA	NA	NA	NA

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.
2 Field duplicates will be collected at a frequency of one per every 10 samples per SWMU.
3 Trip blanks will be submitted for analysis at a frequency of one per cooler containing samples for volatile analysis. Because the number of sample coolers shipped may vary, the number of trip blanks is estimated.
4 Rinsate blanks will be collected at a frequency of one per every 10 samples, with a minimum of one per day of sampling, per sampling device or instrument. These amounts are estimates and may vary.
5 Ambient condition blanks are optional samples that may be collected based on the judgment of the FOL.
6 Matrix spikes are collected for all organic and inorganic parameters at a frequency of one per every 20 field samples per matrix.
7 Matrix spike duplicates are collected for all organic parameters. Lab duplicates are analyzed for inorganic parameters in lieu of matrix spike duplicates. Matrix spike duplicates are collected at a frequency of one per every 20 field samples per matrix.

TABLE 3-4

SUMMARY OF GROUND WATER SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 3

	Parameter [1]	Volatiles		Semivolatiles			Explosives		Metals [2]		Miscellaneous	
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Mercury Total SW-846 7470A	Nitrate + Nitrite as Nitrate EPA 353.2	Ammonia EPA 350.1
SWMU 12												
12MWT01							1		1	1	1	1
12MWT02							1		1	1	1	1
12MWT03							1		1	1	1	1
12MWT04							1		1	1	1	1
12MWT05							1		1	1	1	1
12MWT06							1		1	1	1	1
12MWT07							1		1	1	1	1
12MWT08							1		1	1	1	1
12MWT09							1		1	1	1	1
12MWT10							1		1	1	1	1
12MWT11							1		1	1	1	1
12MWT12							1		1	1	1	1
12MWT13							1		1	1	1	1
12MWT14							1		1	1	1	1
12MWT15							1		1	1	1	1
12MWT16							1		1	1	1	1
12MWT17							1		1	1	1	1
12MWT18							1		1	1	1	1
12MWT19							1		1	1	1	1
12MWT20							1		1	1	1	1
12MWT21							1		1	1	1	1
12MWT22							1		1	1	1	1
12MWT23							1		1	1	1	1
12MWT24							1		1	1	1	1
12MWT25							1		1	1	1	1
12MWT26							1		1	1	1	1
Total Samples SWMU 12		0	0	0	0	0	26	0	26	26	26	26
SWMU 13												

TABLE 3-4

SUMMARY OF GROUND WATER SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 3

	Parameter [1]	Volatiles		Semivolatiles			Explosives		Metals [2]		Miscellaneous	
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Mercury Total SW-846 7470A	Nitrate + Nitrite as Nitrate EPA 353.2	Ammonia EPA 350.1
13MWT01							1		1	1	1	1
13MWT02							1		1	1	1	1
13MWT03							1		1	1	1	1
13MWT04							1		1	1	1	1
13MWT05							1		1	1	1	1
13MWT06							1		1	1	1	1
13MWT07							1		1	1	1	1
13MWT08							1		1	1	1	1
13MWT09		1	1	1	1	1	1		1	1	1	1
13MWT10							1		1	1	1	1
13MWT11							1		1	1	1	1
13MWT12							1		1	1	1	1
13MWT13							1		1	1	1	1
13MWT14							1		1	1	1	1
13MWT15							1		1	1	1	1
13MWT16							1		1	1	1	1
13MWT17							1		1	1	1	1
13MWT18							1		1	1	1	1
13MWT19							1		1	1	1	1
13MWT20							1		1	1	1	1
13MWT21							1		1	1	1	1
13MWT22							1		1	1	1	1
13MWT23							1		1	1	1	1
13MWT24							1		1	1	1	1
13MWT25							1		1	1	1	1
13MWT26							1		1	1	1	1
Total Samples SWMU 13		1	1	1	1	1	26	0	26	26	26	26
SWMU 16												
16MWT01		1	1	1	1	1	1	1	1	1	1	1

TABLE 3-4

SUMMARY OF GROUND WATER SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 3 OF 3

	Parameter [1]	Volatiles		Semivolatiles			Explosives		Metals [2]		Miscellaneous	
Description	Sample ID	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Mercury Total SW-846 7470A	Nitrate + Nitrite as Nitrate EPA 353.2	Ammonia EPA 350.1
16MWT02		1	1	1	1	1	1	1	1	1	1	1
16MWT03		1	1	1	1	1	1	1	1	1	1	1
16MWT04		1	1	1	1	1	1	1	1	1	1	1
16MWT05		1	1	1	1	1	1	1	1	1	1	1
WES-14-01-83		1	1	1	1	1	1	1	1	1	1	1
WES-14-02-83		1	1	1	1	1	1	1	1	1	1	1
WES-14-03-83		1	1	1	1	1	1	1	1	1	1	1
WES-14-04-83		1	1	1	1	1	1	1	1	1	1	1
WES-14-05-83		1	1	1	1	1	1	1	1	1	1	1
Total Samples SWMU 16		10	10	10	10	10	10	10	10	10	10	10
SWMU 19 [3]												
OTA												
RR												
Annex												
Total Samples SWMU 19		0	0	0	0	0	0	0	0	0		

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.
2 Dissolved metals will be performed on GW samples with NTU > 10.
3 Up to 30 wells may be installed at SWMU 19 during Round 3 based on results of Rounds 1 and 2 sampling.

TABLE 3-5

SUMMARY OF GROUND WATER ANALYSES AND QUALITY CONTROL SAMPLES
CTO 166
NSWC CRANE, CRANE, INDIANA

Parameter [1]	Volatiles		Semivolatiles			Explosives		Metals		Miscellaneous	
Sample Type	SW-846 8260B	SW-846 8015B	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Mercury Total SW-846 7470A	Nitrate + Nitrite as Nitrate EPA 353.2	Ammonia EPA 350.1
Samples SWMU 12	0	0	0	0	0	26	0	26	26	26	26
Samples SWMU 13	1	1	1	1	1	26	0	26	26	26	26
Samples SWMU 16	10	10	10	10	10	10	10	10	10	10	0
Samples SWMU 19	0	0	0	0	0	0	0	0	0	0	0
Total Samples All SWMUs	**11**	**11**	**11**	**11**	**11**	**62**	**10**	**62**	**62**	**62**	**52**
Field Duplicates [2]	2	2	2	2	2	7	1	7	7	7	3
Trip Blanks [3]	1	1	NA	NA	NA	NA	NA	NA	NA	NA	NA
Rinsate Blanks [4]	2	2	2	2	2	7	1	7	7	7	3
Ambient Condition Blanks [5]	1	1	1	1	1	1	1	1	1	1	1
Matrix Spikes [6]	1	1	1	1	1	4	1	4	4	4	3
Matrix Spike Duplicates [7]	1	1	1	1	1	4	1	NA	NA	NA	NA

1 See Table 1-6 of this QAPP for specific analysis requirements and analyte lists.
2 Field duplicates will be collected at a frequency of one per every 10 samples per SWMU.
3 Trip blanks will be submitted for analysis at a frequency of one per cooler containing samples for volatile analysis. Because the number of sample coolers shipped may vary, the number of trip blanks are estimated.
4 Rinsate blanks will be collected at a frequency of one per every 10 samples, with a minimum of one per day of sampling, per sampling device or instrument. These amounts are estimates and may vary.
5 Amibient condition blanks are optional samples that may be collected based on the judgment of the FOL.
6 Matrix spikes are collected for all organic and inorganic parameters at a frequency of one per every 20 field samples per matrix.
7 Matrix spike duplicates are collected for all organic parameters. Lab duplicates are analyzed for inorganic parameters in lieu of matrix spike duplicates. Matrix spike duplicates are collected at a frequency of one per every 20 field samples per matrix.

TABLE 3-6

PHYSICAL CHARACTERISTICS OF POTENTIAL MONITORING WELLS
SWMU 12
NSWC CRANE, CRANE, INDIANA

Monitoring Well Number	Approximate Ground Surface Elevation (feet NGVD)	Approximate Total Depth (feet bgs)	Screen Length (feet)	Approximate Desired Well Screen Elevation[1]	
				Top (feet NGVD)	Bottom (feet NGVD)
12MWT01	TBD	TBD	10	TBD	TBD
12MWT02	TBD	TBD	10	TBD	TBD
12MWT03	TBD	TBD	10	TBD	TBD
12MWT04	TBD	TBD	10	TBD	TBD
12MWT05	TBD	TBD	10	TBD	TBD
12MWT06	TBD	TBD	10	TBD	TBD
12MWT07	TBD	TBD	10	TBD	TBD
12MWT08	TBD	TBD	10	TBD	TBD
12MWT09	TBD	TBD	10	TBD	TBD
12MWT10	TBD	TBD	10	TBD	TBD
12MWT11	TBD	TBD	10	TBD	TBD
12MWT12	TBD	TBD	10	TBD	TBD
12MWT13	TBD	TBD	10	TBD	TBD
12MWT14	TBD	TBD	10	TBD	TBD
12MWT15	TBD	TBD	10	TBD	TBD
12MWT16	TBD	TBD	10	TBD	TBD
12MWT17	TBD	TBD	10	TBD	TBD
12MWT18	TBD	TBD	10	TBD	TBD
12MWT19	TBD	TBD	10	TBD	TBD
12MWT20	TBD	TBD	10	TBD	TBD
12MWT21	TBD	TBD	10	TBD	TBD
12MWT22	TBD	TBD	10	TBD	TBD
12MWT23	TBD	TBD	10	TBD	TBD
12MWT24	TBD	TBD	10	TBD	TBD
12MWT25	TBD	TBD	10	TBD	TBD
12MWT26	TBD	TBD	10	TBD	TBD

NGVD - National Geodetic Vertical Datum.
TBD - To Be Determined based on the exact well location and the depth that shallow ground water is encountered during drilling.
bgs - below ground surface.

1 Elevations will vary depending on the soil and rock types and fracture distributions encountered in each boring, with the intent of monitoring similar hydrogeologic zones.

TABLE 3-7

PHYSICAL CHARACTERISTICS OF POTENTIAL MONITORING WELLS
SWMU 13
NSWC CRANE, CRANE, INDIANA

Monitoring Well Number	Approximate Ground Surface Elevation (feet NGVD)	Approximate Total Depth (feet bgs)	Screen Length (feet)	Approximate Desired Well Screen Elevation[1]	
				Top (feet NGVD)	Bottom (feet NGVD)
13MWT01	TBD	TBD	10	TBD	TBD
13MWT02	TBD	TBD	10	TBD	TBD
13MWT03	TBD	TBD	10	TBD	TBD
13MWT04	TBD	TBD	10	TBD	TBD
13MWT05	TBD	TBD	10	TBD	TBD
13MWT06	TBD	TBD	10	TBD	TBD
13MWT07	TBD	TBD	10	TBD	TBD
13MWT08	TBD	TBD	10	TBD	TBD
13MWT09	TBD	TBD	10	TBD	TBD
13MWT10	TBD	TBD	10	TBD	TBD
13MWT11	TBD	TBD	10	TBD	TBD
13MWT12	TBD	TBD	10	TBD	TBD
13MWT13	TBD	TBD	10	TBD	TBD
13MWT14	TBD	TBD	10	TBD	TBD
13MWT15	TBD	TBD	10	TBD	TBD
13MWT16	TBD	TBD	10	TBD	TBD
13MWT17	TBD	TBD	10	TBD	TBD
13MWT18	TBD	TBD	10	TBD	TBD
13MWT19	TBD	TBD	10	TBD	TBD
13MWT20	TBD	TBD	10	TBD	TBD
13MWT21	TBD	TBD	10	TBD	TBD
13MWT22	TBD	TBD	10	TBD	TBD
13MWT23	TBD	TBD	10	TBD	TBD
13MWT24	TBD	TBD	10	TBD	TBD
13MWT25	TBD	TBD	10	TBD	TBD
13MWT26	TBD	TBD	10	TBD	TBD

NGVD - National Geodetic Vertical Datum.
TBD - To Be Determined based on the exact well location and the depth that shallow ground water is encountered during drilling.
Bgs - below ground surface.

1 Elevations will vary depending on the soil and rock types and fracture distributions encountered in each boring, with the intent of monitoring similar hydrogeologic zones.

TABLE 3-8

SUMMARY OF MONITORING WELL CONSTRUCTION DETAILS AT THE CAST HIGH EXPLOSIVES FILL (1)
NSWC CRANE, CRANE, INDIANA

Monitoring Well Number	Year Installed	Top of Pipe Elevation (feet-MSL)	Ground Surface Elevation (feet-MSL)	Well Screen Interval		Well Screen Interval		Ground water Depth & Elevation		
				Depth to Top (feet-BGS)	Depth to Bottom (feet-BGS)	Elevation of Top (feet)	Elevation of Bottom (feet)	Date Measured	Depth to Water (feet - TOP)	Ground water Elevation (feet-MSL)
WES-14-1-83	1983	767.37	764.37	35.80	40.47	728.57	723.90	10/4/83	36.35	731.02
WES-14-2-83	1983	767.31	764.31	11.35	20.39	752.96	743.92	10/4/83	15.59	751.72
WES-14-3-83	1983	764.65	761.65	20.88	29.99	740.77	731.66	10/4/83	22.75	741.90
WES-14-4-83	1983	763.73	760.73	31.38	40.47	729.35	720.26	10/4/83	31.05	732.68
WES-14-5-83	1983	770.10	767.10	35.50	44.75	731.60	722.35	10/4/83	37.75	732.35

1 All data were obtained from RCRA Facility Investigation Phase I Environmental Monitoring Reports Solid Waste Management Units #15/06,#14/00, and #16/16 Naval Surface Warfare Center, Crane, Indiana.

MSL - Mean Sea Level.

TABLE 3-9

PHYSICAL CHARACTERISTICS OF POTENTIAL MONITORING WELLS
SWMU 16
NSWC CRANE, CRANE, INDIANA(1)

Monitoring Well Number	Approximate Ground Surface Elevation (feet NGVD)	Approximate Total Depth (feet bgs)	Screen Length (feet)	Approximate Desired Well Screen Elevation(2)	
				Top	Bottom
				(feet NGVD)	(feet NGVD)
16MWT01	770	45	10	725	715
16MWT02	770	45	10	725	715
16MWT03	770	45	10	725	715
16MWT04	770	45	10	725	715
16MWT05	770	45	10	725	715

NGVD - National Geodetic Vertical Datum.
Bgs - below ground surface.

1 Exact elevations, depths, and screen intervals will be determined based on the exact well location and the depth that shallow ground water is encountered during drilling.

2 Elevations will vary depending on the soil and rock types and fracture distributions encountered in each boring, with the intent of monitoring similar hydrogeologic zones.

TABLE 3-10

PHYSICAL CHARACTERISTICS OF POTENTIAL MONITORING WELLS
SWMU 19
NSWC CRANE, CRANE, INDIANA

Monitoring Well Number	Approximate Ground Surface Elevation (feet NGVD)	Approximate Total Depth (feet bgs)	Screen Length (feet)	Approximate Desired Well Screen Elevation[1]	
				Top (feet NGVD)	Bottom (feet NGVD)
19MWT01	TBD	TBD	10	TBD	TBD
19MWT02	TBD	TBD	10	TBD	TBD
19MWT03	TBD	TBD	10	TBD	TBD
19MWT04	TBD	TBD	10	TBD	TBD
19MWT05	TBD	TBD	10	TBD	TBD
19MWT06	TBD	TBD	10	TBD	TBD
19MWT07	TBD	TBD	10	TBD	TBD
19MWT08	TBD	TBD	10	TBD	TBD
19MWT09	TBD	TBD	10	TBD	TBD
19MWT10	TBD	TBD	10	TBD	TBD
19MWT11	TBD	TBD	10	TBD	TBD
19MWT12	TBD	TBD	10	TBD	TBD
19MWT13	TBD	TBD	10	TBD	TBD
19MWT14	TBD	TBD	10	TBD	TBD
19MWT15	TBD	TBD	10	TBD	TBD
19MWT16	TBD	TBD	10	TBD	TBD
19MWT17	TBD	TBD	10	TBD	TBD
19MWT18	TBD	TBD	10	TBD	TBD
19MWT19	TBD	TBD	10	TBD	TBD
19MWT20	TBD	TBD	10	TBD	TBD
19MWT21	TBD	TBD	10	TBD	TBD
19MWT22	TBD	TBD	10	TBD	TBD
19MWT23	TBD	TBD	10	TBD	TBD
19MWT24	TBD	TBD	10	TBD	TBD
19MWT25	TBD	TBD	10	TBD	TBD
19MWT26	TBD	TBD	10	TBD	TBD
19MWT27	TBD	TBD	10	TBD	TBD
19MWT28	TBD	TBD	10	TBD	TBD
19MWT29	TBD	TBD	10	TBD	TBD
19MWT30	TBD	TBD	10	TBD	TBD

NGVD - National Geodetic Vertical Datum.
TBD - To Be Determined based on the exact well location and the depth that shallow groundwater is encountered during drilling.
Bgs - below ground surface.

1 Elevations will vary depending on the soil and rock types and fracture distributions encountered in each boring, with the intent of monitoring similar hydrogeologic zones.

TABLE 3-11
SUMMARY OF SURFACE WATER SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 3

Parameter [1]	Semivolatiles			Explosives		Metals				Miscellaneous		
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Metals Dissolved SW-846 6020	Mercury Total SW-846 7470A	Mercury Dissolved SW-846 7470A	Perchlorate	Nitrate + Nitrite as Nitrate	Ammonia
SWMU 12												
12SW01				1		1	1	1	1		1	1
12SW02				1		1	1	1	1		1	1
12SW03				1		1	1	1	1		1	1
12SW04				1		1	1	1	1		1	1
12SW05				1		1	1	1	1		1	1
12SW06				1		1	1	1	1		1	1
12SW07				1		1	1	1	1		1	1
12SW08				1		1	1	1	1		1	1
12SW09	1	1	1	1		1	1	1	1		1	1
12SW10				1		1	1	1	1		1	1
12SW11				1		1	1	1	1		1	1
12SW12				1		1	1	1	1		1	1
12SW13				1		1	1	1	1		1	1
12SW14				1		1	1	1	1		1	1
12SW15				1		1	1	1	1		1	1
Total Samples SWMU 12	1	1	1	15	0	15	15	15	15	0	15	15

TABLE 3-11
SUMMARY OF SURFACE WATER SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 3

Parameter [1]	Semivolatiles			Explosives		Metals				Miscellaneous		
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Metals Dissolved SW-846 6020	Mercury Total SW-846 7470A	Mercury Dissolved SW-846 7470A	Perchlorate	Nitrate + Nitrite as Nitrate	Ammonia
SWMU 13												
13SW01				1		1	1	1	1		1	1
13SW02				1		1	1	1	1		1	1
13SW03				1		1	1	1	1		1	1
13SW04	1	1	1	1		1	1	1	1		1	1
13SW05	1	1	1	1		1	1	1	1		1	1
13SW06	1	1	1	1		1	1	1	1		1	1
13SW07				1		1	1	1	1		1	1
13SW08				1		1	1	1	1		1	1
13SW09				1		1	1	1	1		1	1
13SW10				1		1	1	1	1		1	1
13SW11				1		1	1	1	1		1	1
13SW12				1		1	1	1	1		1	1
13SW13				1		1	1	1	1		1	1
Total Samples SWMU 13	3	3	3	13	0	13	13	13	13	0	13	13
SWMU 16												
16SW01	1	1	1	1	1	1	1	1	1		1	
16SW02	1	1	1	1	1	1	1	1	1		1	
16SW03	1	1	1	1	1	1	1	1	1		1	
16SW04	1	1	1	1	1	1	1	1	1		1	
16SW05	1	1	1	1	1	1	1	1	1		1	
16SW06	1	1	1	1	1	1	1	1	1		1	
16SW07	1	1	1	1	1	1	1	1	1		1	
Total Samples SWMU 16	7	7	7	7	7	7	7	7	7	0	7	0

TABLE 3-11
SUMMARY OF SURFACE WATER SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 3 OF 3

Parameter [1]	Semivolatiles			Explosives		Metals				Miscellaneous		
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Metals Dissolved SW-846 6020	Mercury Total SW-846 7470A	Mercury Dissolved SW-846 7470A	Perchlorate	Nitrate + Nitrite as Nitrate	Ammonia
SWMU 19												
OTA												
19SW01				1		1	1	1	1		1	
19SW02				1		1	1	1	1		1	
19SW03				1		1	1	1	1		1	
19SW04				1		1	1	1	1		1	
19SW05				1		1	1	1	1		1	
19SW06				1		1	1	1	1		1	
19SW07				1		1	1	1	1		1	
19SW08				1		1	1	1	1		1	
19SW09				1		1	1	1	1		1	
19SW10				1		1	1	1	1		1	
19SW11				1		1	1	1	1		1	
19SW12				1		1	1	1	1		1	
19SW13				1		1	1	1	1		1	
19SW14				1		1	1	1	1		1	
19SW15				1		1	1	1	1		1	
19SW16				1		1	1	1	1		1	
19SW17				1		1	1	1	1		1	
19SW18				1		1	1	1	1		1	
19SW26				1		1	1	1	1		1	
19SW27				1		1	1	1	1		1	
RR												
19SW22				1		1	1	1	1	1	1	
19SW23				1		1	1	1	1	1	1	
19SW24				1		1	1	1	1	1	1	
19SW25				1		1	1	1	1	1	1	
Annex												
19SW19				1		1	1	1	1		1	
19SW20				1		1	1	1	1		1	
19SW21				1		1	1	1	1		1	
Total Samples SWMU 19	0	0	0	27	0	27	27	27	27	4	27	0

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.

TABLE 3-12

SUMMARY OF SURFACE WATER ANALYSES AND QUALITY CONTROL SAMPLES
CTO 166
NSWC CRANE, CRANE, INDIANA

Parameter [1]	Semivolatiles			Explosives		Metals				Miscellaneous		
Sample Type	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals Total SW-846 6020	Metals Dissolved SW-846 6020	Mercury Total SW-846 7470A	Mercury Dissolved SW-846 7470A	Perchlorate	Nitrate + Nitrite as Nitrate	Ammonia
Samples SWMU 12	1	1	1	15	0	15	15	15	15	0	15	15
Samples SWMU 13	3	3	3	13	0	13	13	13	13	0	13	13
Samples SWMU 16	7	7	7	7	7	7	7	7	7	0	7	0
Samples SWMU 19	0	0	0	27	0	27	27	27	27	4	27	0
Total Samples All SWMUs	**11**	**11**	**11**	**62**	**7**	**62**	**62**	**62**	**62**	**4**	**62**	**28**
Field Duplicates [2]	2	2	2	7	1	7	7	7	7	1	7	3
Trip Blanks [3]	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA	NA
Rinsate Blanks [4]	0	0	0	0	0	0	0	0	0	0	0	0
Ambient Condition Blanks [5]	1	1	1	1	1	1	1	1	1	1	1	1
Matrix Spikes [6]	1	1	1	4	1	4	4	4	4	1	4	2
Matrix Spike Duplicates [7]	1	1	1	4	1	NA	NA	NA	NA	NA	NA	NA

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.
2 Field duplicates will be collected at a frequency of one per every 10 samples per SWMU.
3 Trip blanks will be submitted for analysis at a frequency of one per cooler containing samples for volatile analysis. Because the number of sample coolers shipped may vary, the number of trip blanks is estimated.
4 Environmental samples will be collected by directly filling the sample bottle. Therefore, no rinsate blanks are required for this matrix.
5 Ambient condition blanks are optional samples that may be collected based on the judgment of the FOL.
6 Matrix spikes are collected for all organic and inorganic parameters at a frequency of one per every 20 field samples per matrix.
7 Matrix spike duplicates are collected for all organic parameters. Lab duplicates are analyzed for inorganic parameters in lieu of matrix spike duplicates. Matrix spike duplicates are collected at a frequency of one per every 20 field samples per matrix.

TABLE 3-13

SUMMARY OF SEDIMENT SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 4

Parameter [1]	Semivolatiles			Explosives		Metals		Miscellaneous				
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals SW-846 6020	Mercury SW-846 7471A	Perchlorate	CEC	AVS/SEM	TOC	pH
SWMU 12												
12SD01				1		1	1		1	1	1	1
12SD02				1		1	1					
12SD03				1		1	1					
12SD04				1		1	1		1	1	1	1
12SD05				1		1	1					
12SD06				1		1	1					
12SD07				1		1	1		1	1	1	1
12SD08				1		1	1					
12SD09	1	1	1	1		1	1					
12SD10				1		1	1		1	1	1	1
12SD11				1		1	1					
12SD12				1		1	1					
12SD13				1		1	1		1	1	1	1
12SD14				1		1	1					
12SD15				1		1	1					
12SD16				1		1	1		1	1	1	1
12SD17				1		1	1					
12SD18	1	1	1	1		1	1					
12SD19				1		1	1		1	1	1	1
12SD20				1		1	1					
12SD21				1		1	1					
12SD22				1		1	1		1	1	1	1
12SD23				1		1	1					
12SD24 (TBD)	1	1	1	1		1	1					
Total Samples SWMU 12	3	3	3	24	0	24	24	0	8	8	8	8

TABLE 3-13

SUMMARY OF SEDIMENT SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 4

Parameter [1]	Semivolatiles			Explosives		Metals		Miscellaneous				
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals SW-846 6020	Mercury SW-846 7471A	Perchlorate	CEC	AVS/SEM	TOC	pH
SWMU 13												
13SD01				1		1	1					
13SD02				1		1	1		1	1	1	1
13SD03				1		1	1					
13SD04	1	1	1	1		1	1					
13SD05	1	1	1	1		1	1		1	1	1	1
13SD06	1	1	1	1		1	1					
13SD07				1		1	1					
13SD08				1		1	1		1	1	1	1
13SD09				1		1	1					
13SD10				1		1	1					
13SD11				1		1	1		1	1	1	1
13SD12				1		1	1					
13SD13				1		1	1					
13SD14				1		1	1		1	1	1	1
13SD15				1		1	1					
13SD16				1		1	1					
13SD17				1		1	1		1	1	1	1
13SD18				1		1	1					
13SD19				1		1	1					
13SD20				1		1	1		1	1	1	1
13SD21				1		1	1					
13SD22				1		1	1					
Total Samples SWMU 13	3	3	3	22	0	22	22	0	7	7	7	7
SWMU 16												
16SD01	1	1	1	1	1	1	1					
16SD02	1	1	1	1	1	1	1		1	1	1	1
16SD03	1	1	1	1	1	1	1					
16SD04	1	1	1	1	1	1	1					
16SD05	1	1	1	1	1	1	1					
16SD06	1	1	1	1	1	1	1		1	1	1	1

TABLE 3-13

SUMMARY OF SEDIMENT SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 3 OF 4

Parameter [1]	Semivolatiles			Explosives		Metals		Miscellaneous				
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals SW-846 6020	Mercury SW-846 7471A	Perchlorate	CEC	AVS/SEM	TOC	pH
16SD07	1	1	1	1	1	1	1					
16SD08	1	1	1	1	1	1	1					
16SD09	1	1	1	1	1	1	1		1	1	1	1
16SD10	1	1	1	1	1	1	1					
16SD11	1	1	1	1	1	1	1					
Total Samples SWMU 16	11	11	11	11	11	11	11	0	3	3	3	3
SWMU 19												
OTA												
19SD01				1		1	1					
19SD02				1		1	1		1	1	1	1
19SD03				1		1	1					
19SD04				1		1	1					
19SD05				1		1	1					
19SD06				1		1	1		1	1	1	1
19SD07				1		1	1					
19SD08				1		1	1					
19SD09				1		1	1		1	1	1	1
19SD10				1		1	1					
19SD11				1		1	1		1	1	1	1
19SD12				1		1	1					
19SD13				1		1	1		1	1	1	1
19SD14				1		1	1					
19SD15				1		1	1					
19SD16				1		1	1					
19SD17				1		1	1					
19SD18				1		1	1		1	1	1	1
19SD26				1		1	1					
19SD27				1		1	1		1	1	1	1
Rocket Range												
19SD22				1		1	1	1				
19SD23				1		1	1	1				

TABLE 3-13

SUMMARY OF SEDIMENT SAMPLES AND LABORATORY ANALYSES
CTO 166
NSWC CRANE, CRANE, INDIANA
PAGE 4 OF 4

Parameter [1]	Semivolatiles			Explosives		Metals		Miscellaneous				
Description	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals SW-846 6020	Mercury SW-846 7471A	Perchlorate	CEC	AVS/SEM	TOC	pH
19SD24				1		1	1	1	1	1	1	1
19SD25				1		1	1	1				
Annex												
19SD19				1		1	1					
19SD20				1		1	1		1	1	1	1
19SD21				1		1	1					
Total Samples SWMU 19	0	0	0	27	0	27	27	4	9	9	9	9

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.
TBD - This sample will be collected in the drain sump of the UAP, if the drain sump can be found and contains a sufficient volume of sediment for sampling.

TABLE 3-14

SUMMARY OF SEDIMENT ANALYSES AND QUALITY CONTROL SAMPLES
CTO 166
NSWC CRANE, CRANE, INDIANA

Parameters [1]	Semivolatiles			Explosives		Metals		Miscellaneous				
Sample Type	SW-846 8270C	Selected Ion Monitoring	SW-846 8151A	SW-846 8330	Picrates	Metals SW-846 6020	Mercury SW-846 7471A	Perchlorate	CEC	AVS/SEM	TOC	pH
Samples SWMU 12	3	3	3	24	0	24	24	0	8	8	8	8
Samples SWMU 13	3	3	3	22	0	22	22	0	7	7	7	7
Samples SWMU 16	11	11	11	11	11	11	11	0	3	3	3	3
Samples SWMU 19	0	0	0	27	0	27	27	4	9	9	9	9
Total Samples All SWMUs	**17**	**17**	**17**	**84**	**11**	**84**	**84**	**4**	**27**	**27**	**27**	**27**
Field Duplicates [2]	2	2	2	9	2	9	9	1	3	3	3	3
Rinsate Blanks [3]	2	2	2	9	2	9	9	1	NA	NA	3	NA
Ambient Condition Blanks [4]	1	1	1	1	1	1	1	1	NA	NA	NA	NA
Matrix Spikes [5]	1	1	1	5	1	5	5	1	NA	NA	2	NA
Matrix Spike Duplicates [6]	1	1	1	5	1	NA	NA	NA	NA	NA	NA	NA

1 See Table 1-7 of this QAPP for specific analysis requirements and analyte lists.
2 Field duplicates will be collected at a frequency of one per every 10 samples per SWMU.
3 Rinsate blanks will be collected at a frequency of one per every 10 samples, with a minimum of one per day of sampling, per sampling device or instrument. These amounts are estimates and may vary.
4 Ambient condition blanks are optional samples that may be collected based on the judgment of the FOL.
5 Matrix spikes are collected for all organic and inorganic parameters at a frequency of one per every 20 field samples per matrix.
6 Matrix spike duplicates are collected for all organic parameters. Lab duplicates are analyzed for inorganic parameters in lieu of matrix spike duplicates. Matrix spike duplicates are collected at a frequency of one per every 20 field samples per matrix.

TABLE 3-15

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR SOILS AND SEDIMENTS CTO 166 NSWC CRANE PAGE 1 OF 2

Parameter	Analyte/Methodology	Sample Container	Container Volume	Preservation	Maximum Holding Time[1]
Explosives	Explosives SW-846 8330	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction 14 days; analysis within 40 days of extraction
	Picric and Picramic Acid Laucks HPLC Method	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction 14 days; analysis within 40 days of extraction
Metals	Metals SW-846 6020, 7471A	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Analysis within 180 days; mercury analysis within 28 days
Volatiles[2]	SW-846 8260B	3 Encore™ samplers	Four 5-gram containers	Cool to 4 °C	48 hours to preservation[3]; 14 days to analysis
	SW-846 8015B	2 Encore™ samplers	Four 5-gram containers	Cool to 4 °C	48 hours to preservation[4]; 7 days to analysis
Semivolatiles	SW-846 8270C	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction 14 days; analysis within 40 days of extraction
	SW-846 8270C Selective Ion Monitoring	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction 14 days; analysis within 40 days of extraction
	PCP and Hexachlorophene SW-846 8151A	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction 14 days; analysis within 40 days of extraction
PCBs	PCBs	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction 14 days; analysis within 40 days of extraction
Dioxins	SW-846 8290	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction within 30 days; analysis within 40 days of extraction.
Dyes	NSWC Crane	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Extraction within 14 days; analysis within 40 days of extraction.
Total Organic Carbon	Llyod Khan	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	28 days
Perchlorate	EPA Method 314	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	28 days to analysis
pH	SW-846 9045C	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	Within 24 hours of sample reciept
Cation Exchange Capacity	SW-846 9081	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	As soon as possible

TABLE 3-15

SUMMARY OF SAMPLE ANALYSIS, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR SOILS AND SEDIMENTS CTO 166, NSWC CRANE PAGE 2 OF 2

Parameter	Analyte/Methodology	Sample Container	Container Volume	Preservation	Maximum Holding Time[1]
Cyanide	SW-846 9012A	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	14 days to analysis
AVS/SEM	Draft EPA Method	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	14 days to analysis
Ammonia	EPA 350.1	Wide-mouth jar, Teflon-lined plastic cap	8 ounce	Cool to 4 °C	28 days to analysis

1 All holding times are from date of collection.

2 Sediment samples collected from areas with no standing water will be collected and containerized in this manner. Sediment samples collected from areas under standing water will be containerized using a 4 ounce clear wide mouth glass jar and cooled to 4°C.

3 One Encore™ extended into 5 mL H_2O and frozen. Second Encore™ frozen as received. Third Encore™ preserved with methanol for medium level analysis.

4 One Encore™ extended into 5 mL H_2O and frozen. Second Encore™ frozen as received.

TABLE 3-16

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR AQUEOUS SAMPLES CTO 166, NSWC CRANE

Parameter	Analyte/Methodology	Sample Container	Container Volume	Preservation[1]	Maximum Holding Time[2]
Explosives	Explosives SW-846 8330	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
	Picric and Picramic Acid Laucks HPLC Method	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
Metals	Total SW-846 6020, 7470A	Polyethylene, plastic cap w/ plastic liner	1000 mL	HNO_3 to pH <2	Within 180 days; mercury within 28 days
	Dissolved SW-846 6020, 7470A	Polyethylene, plastic cap w/ plastic liner	1000 mL	HNO_3 to pH <2	Within 180 days; mercury within 28 days
Volatiles	SW-846 8260B	Glass, black phenolic plastic screw cap, Teflon-lined septum	(6) 40 mL	Cool to 4 °C, dark, zero headspace, HCl to pH <2	14 days to analysis
	SW-846 8015B	Glass, black phenolic plastic screw cap, Teflon-lined septum	(6) 40 mL	Cool to 4 °C, dark, zero headspace, HCl to pH <2	14 days to analysis
Semivolatile	SW-846 8270C	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
	Selective Ion Monitoring SW-846 8270C	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
	PCP and Hexachlorophene SW-846 8151A	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
PCBs	PCBs SW-846 8082	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
Dyes	NSWC Crane	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C, dark	Extraction 7 days; analysis within 40 days of extraction
Dioxin	SW-846 8290	Amber glass, Teflon-lined cap	(2) 1000 mL	Cool to 4 °C	Extraction 30 days; analysis within 45 days of extraction
Perchlorate	EPA Method 314.0	Polyethylene, plastic cap w/ plastic liner	500 mL	Cool to 4 °C, dark	28 days to analysis

TABLE 3-16

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR AQUEOUS SAMPLES CTO 166, NSWC CRANE PAGE 2 OF 2

Parameter	Analyte/Methodology	Sample Container	Container Volume	Preservation[1]	Maximum Holding Time[2]
Total Organic Carbon	SW-846 9060	Amber glass, septa cap	125 mL	Cool to 4 °C, H_2SO_4 to pH < 2	28 days to analysis
Cyanide	SW-846 9012A	Polyethylene, plastic cap w/ plastic liner	500 mL	Cool to 4° C, NaOH to pH > 12	14 days to analysis

Note: Aqueous samples include ground water, surface water, and seeps.

1 HCl = Hydrochloric acid, H_2SO_4 = Sulfuric Acid, HNO_3 = Nitric Acid , NaOH = Sodium Hydroxide.

2 All holding times are from date of collection.

mL = milliliters

TABLE 3-17

NON-CALIBRATION QC SAMPLE USAGE FREQUENCIES, ACCEPTANCE LIMITS AND CORRECTIVE ACTIONS NSWC CRANE, CRANE, INDIANA PAGE 1 OF 2

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Field Duplicate	1 per 10 investigative samples collected.	Aqueous = 30% RPD Soil/Sediment = 50% RPD	Qualify data according to data validation requirements.
Equipment Rinsate Blank	1 per 10 investigative samples collected, with a minimum of one per day of sampling, per non-disposable sampling device/instrument. For pre-cleaned, dedicated, and/or disposable equipment (i.e., disposable plastic trowels, etc.), one rinsate blank will be collected and analyzed at a frequency of one per lot or "batch blank" for a specific equipment type.	< RL (soil and water)	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of data if contamination appears to have adversely affected its usability.
Source Water Blank	1 per each source of water used for sampling equipment decontamination.	< RL (soil and water)	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of data if contamination appears to have adversely affected its usability.
Ambient Condition Blanks	At discretion of FOL.	< RL (soil and water)	Identify source of contamination, if possible. Qualify data according to validation criteria. Qualify use of data if contamination appears to have adversely affected its usability.
Trip Blanks	1 per cooler containing samples for volatile organics analysis.	< RL (soil and water)	Identify source of volatiles contamination, if possible. Qualify data according to validation criteria. Qualify use of data if contamination appears to have adversely affected its usability.
Internal Standard	At least one internal standard per sample for GC/MS analyses.	Retention times stable to ±30 seconds; area counts stable to within factor of 2.	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Laboratory Control Sample	1 per 20 environmental samples per matrix	See Tables 7-2, 7-4, 7-6, 7-8, 7-10, 7-12, 7-14, 7-16	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.

TABLE 3-17

NON-CALIBRATION QC SAMPLE USAGE FREQUENCIES, ACCEPTANCE LIMITS AND CORRECTIVE ACTIONS NSWC CRANE, CRANE, INDIANA PAGE 2 OF 2

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Laboratory Duplicate	1 per 20 environmental samples analyzed for inorganic target analytes	See Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Laboratory Method Blank	1 per 20 environmental samples or per preparation batch, whichever is more frequent	< RL (soil and water)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Matrix Spike*	1 per 20 environmental samples	See Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Matrix Spike Duplicate*	1 per 20 environmental samples analyzed for organic target analytes	See Tables 7-1, 7-3, 7-7, 7-9, 7-11, 7-13, 7-15	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Post-digestion Spike	Only if out-of-control matrix spike exists (metals only)	100 ± 20%	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Surrogate	At least one per sample for organic chromatographic analyses (GC, GC/MS, and HPLC)	See Tables 7-1, 7-3, 7-7, 7-9, 7-11, 7-13, 7-15	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.
Temperature Blank	One blank per sample cooler.	4 ± 2 °C	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.2.

* Matrix spikes and matrix spike duplicates are not analyzed in the field, but additional sample material must be collected in the field to ensure that the laboratory has enough material for spiking and duplicate analysis.

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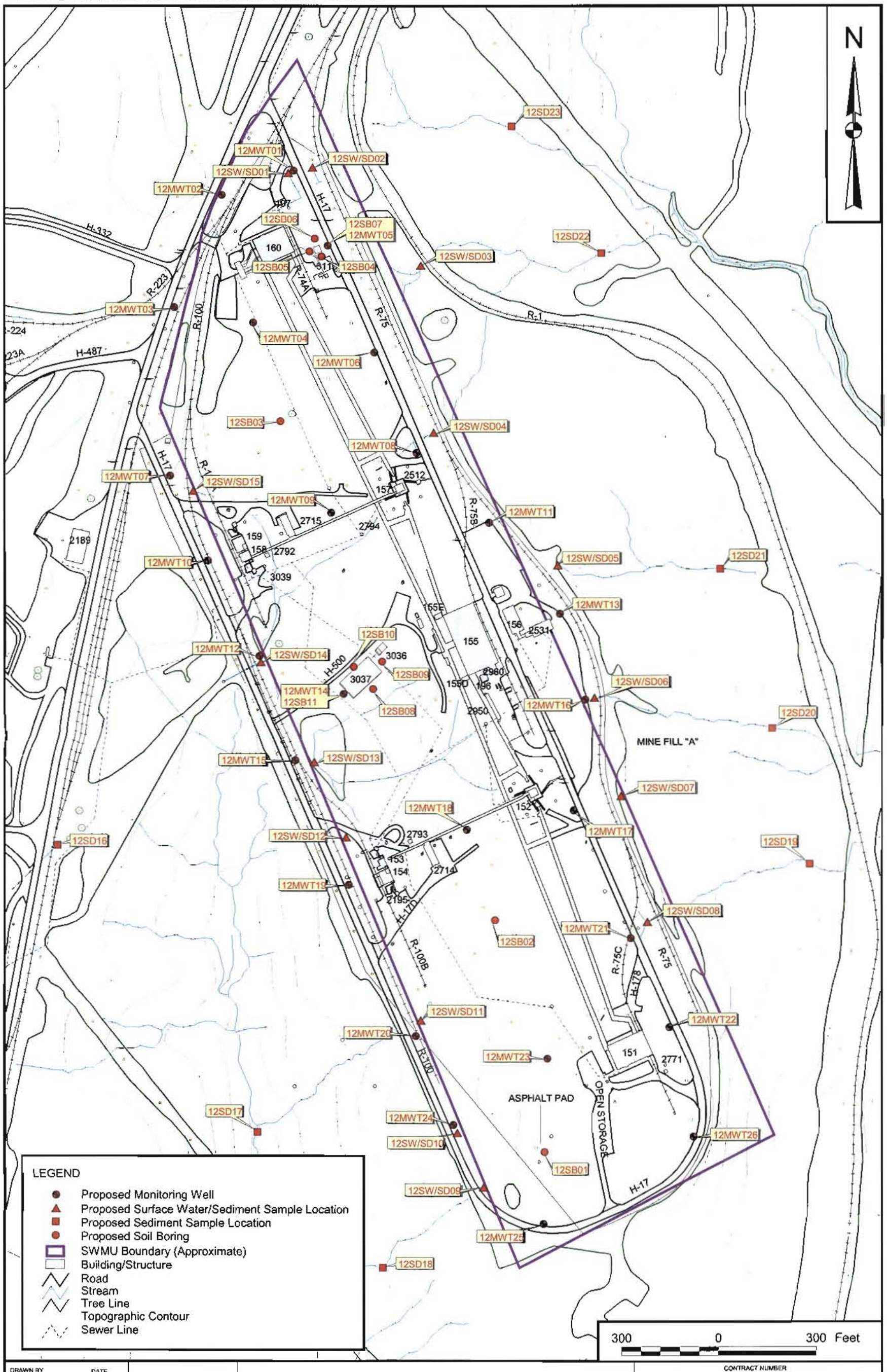
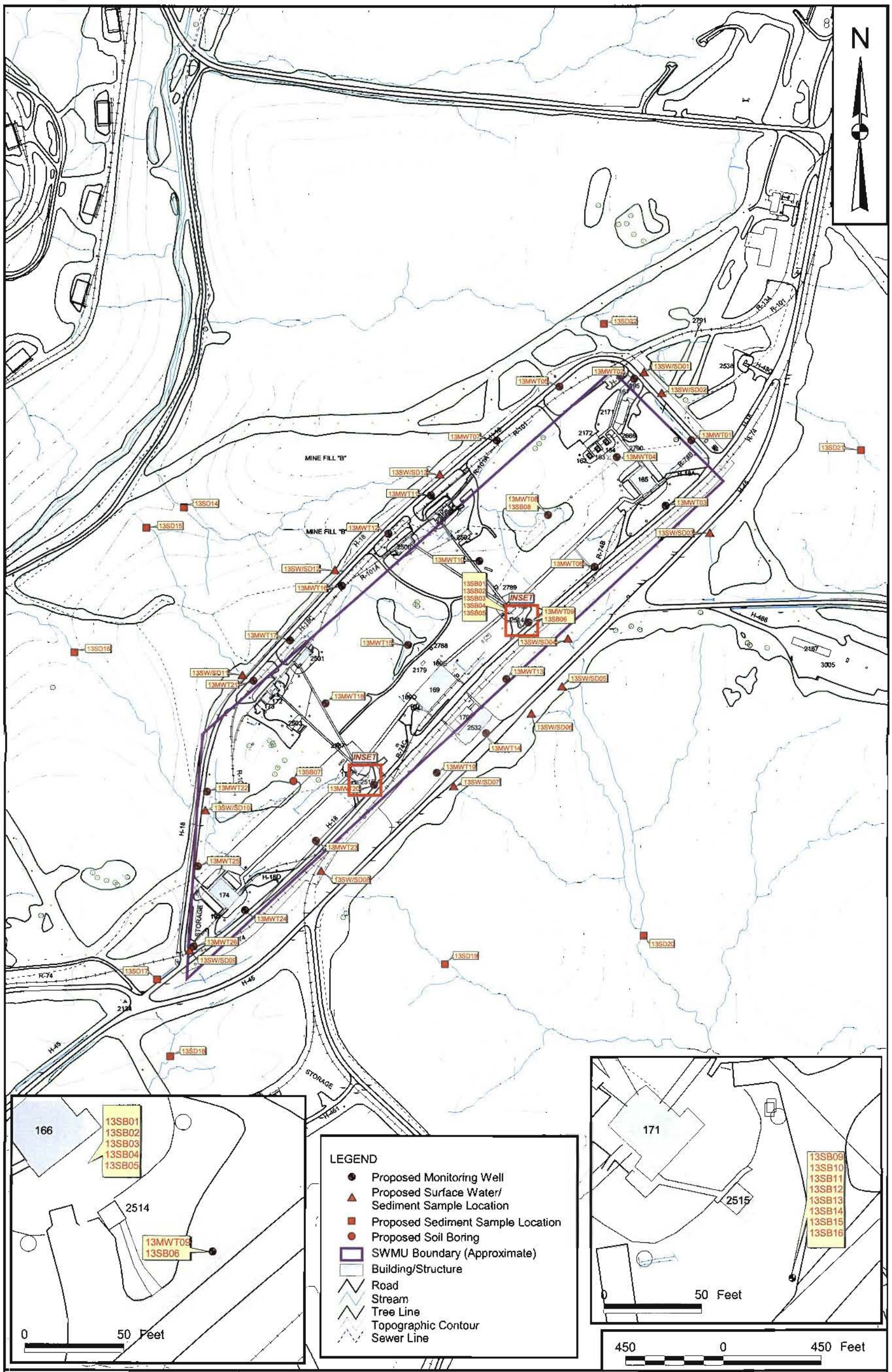
P:\GIS\NSWC_CRANE\APR\MINEFILL_A.APR PROPOSED SAMPLE LOCATIONS 7/23/02 JAL



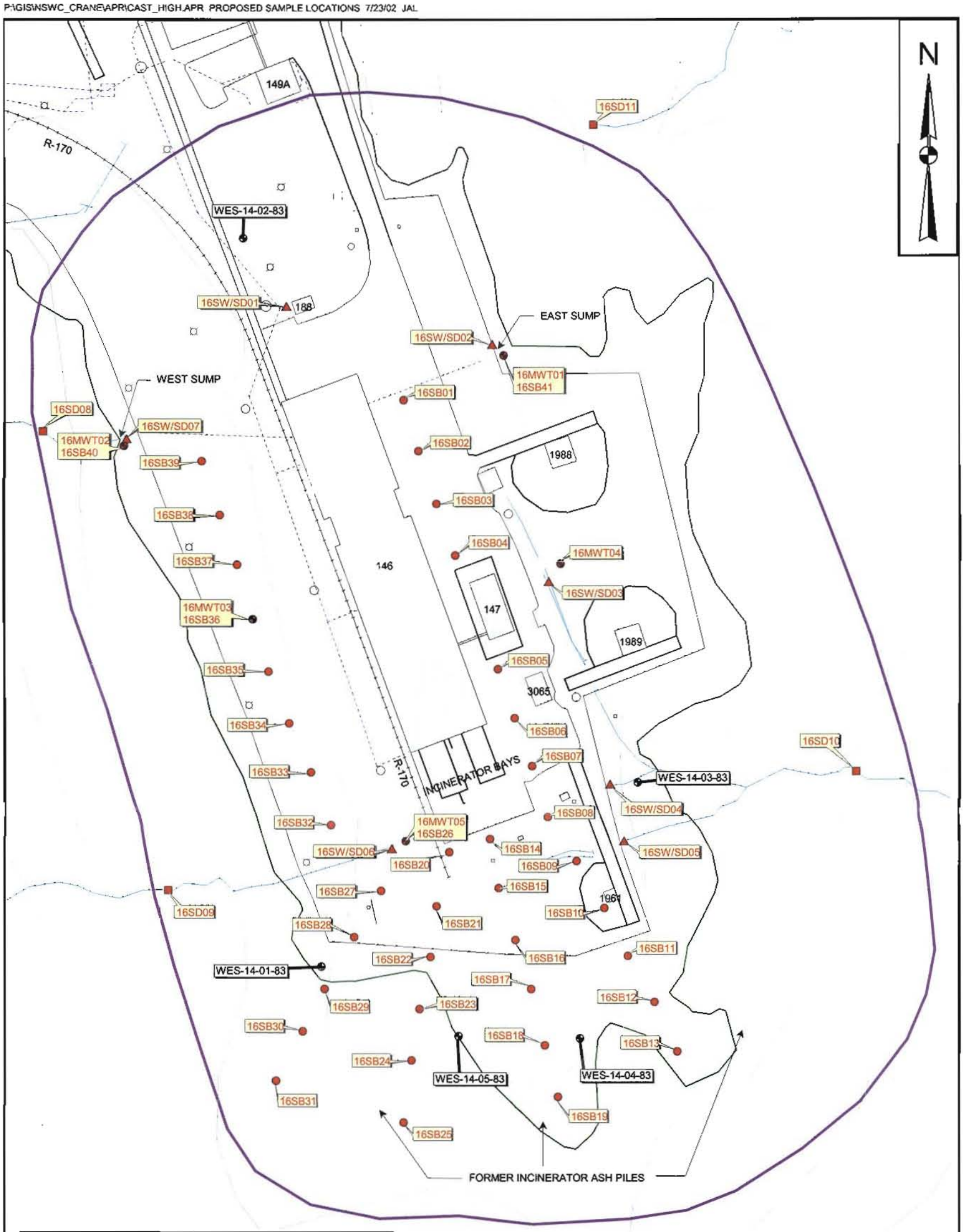
DRAWN BY	DATE		CONTRACT NUMBER
J. LAMEY	8/21/01	PROPOSED SAMPLE LOCATIONS MINE FILL A (SWMU 12/14) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	APPROVED BY / DATE
CHECKED BY	DATE		APPROVED BY / DATE
COST/SCHEDULE-AREA			DRAWING NO FIGURE 3-1 / REV 0
SCALE AS NOTED			

P:\GIS\NSWC_CRANE\APR\MINEFILL_B.APR PROPOSED SAMPLE LOCATIONS 2/19/03 KMP



DRAWN BY	DATE		CONTRACT NUMBER
J LAMEY	8/21/01	PROPOSED SAMPLE LOCATIONS MINE FILL B (SWMU 13/14) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA	—
CHECKED BY	DATE		APPROVED BY — DATE —
COST/SCHEDULE-AREA			APPROVED BY — DATE —
SCALE AS NOTED			DRAWING NO FIGURE 3-2 — REV 0

P:\GIS\NSWC_CRANE\APR\CAST_HIGH.APR PROPOSED SAMPLE LOCATIONS 7/23/02 JAL



LEGEND

- Proposed Monitoring Well
- Proposed Soil Boring
- Proposed Surface Water/Sediment Sample Location
- Proposed Sediment Sample Location
- Existing Monitoring Well
- SWMU Boundary (Approximate)
- Building/Structure
- Road
- Railroad
- Stream
- Tree Line
- Topographic Contour
- Sewer Line

100 0 100 Feet

DRAWN BY	DATE			CONTRACT NUMBER
J LAMEY	8/22/01	PROPOSED SAMPLE LOCATIONS CAST HIGH EXPLOSIVES FILL (SWMU 16/16) NAVAL SURFACE WARFARE CENTER CRANE, INDIANA		APPROVED BY / DATE
CHECKED BY	DATE			APPROVED BY / DATE
COST/SCHEDULE-AREA				DRAWING NO. FIGURE 3-3 / REV 0
SCALE AS NOTED				

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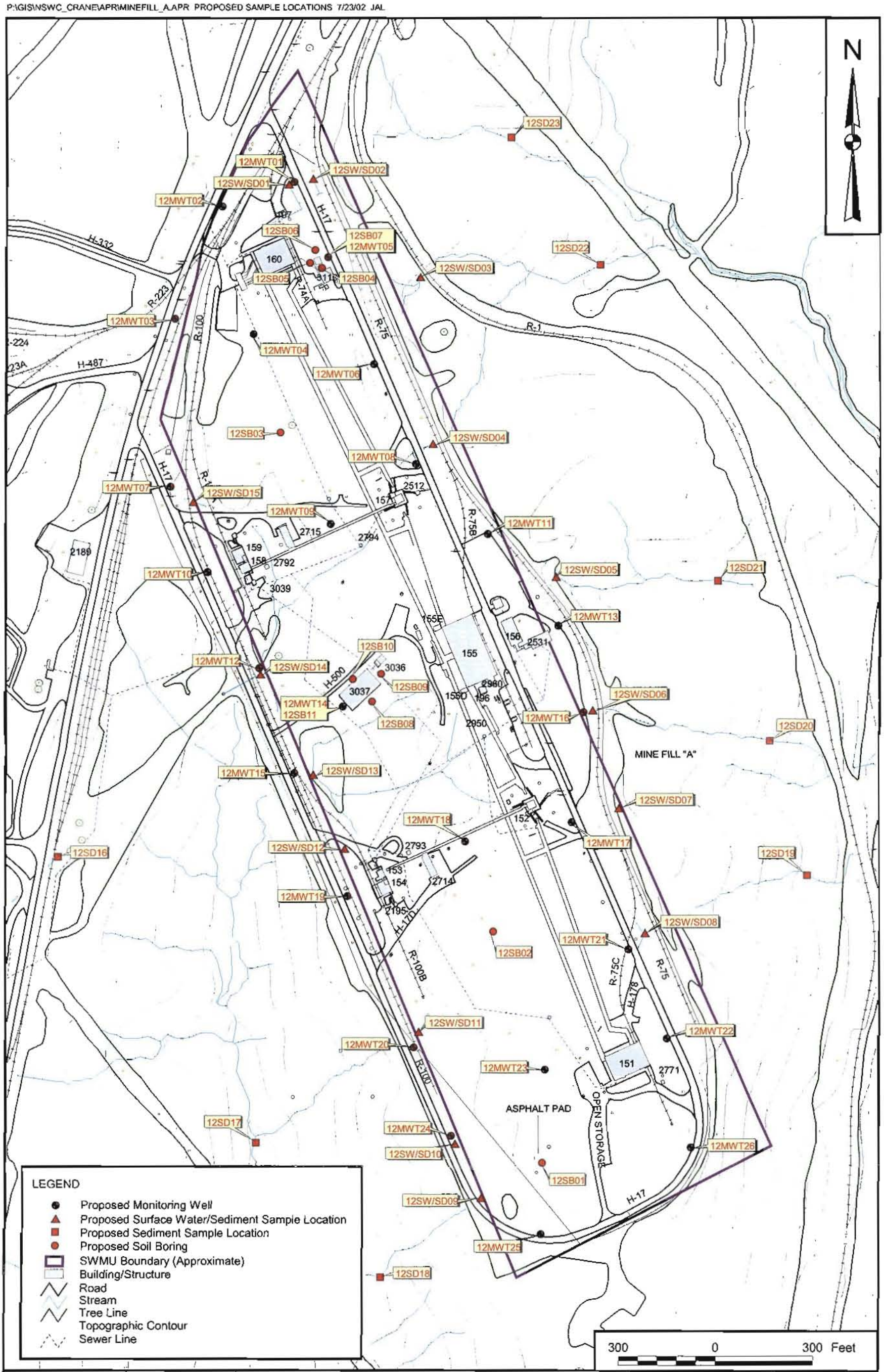

DRAWN BY	DATE
J. LAMEY	8/21/01
CHECKED BY	DATE
COST/SCHEDULE-AREA	
SCALE AS NOTED	

PROPOSED SAMPLE LOCATIONS
MINE FILL A (SWMU 12/14)
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

CONTRACT NUMBER	
APPROVED BY	DATE
APPROVED BY	DATE
DRAWING NO. FIGURE 3-1	REV 0

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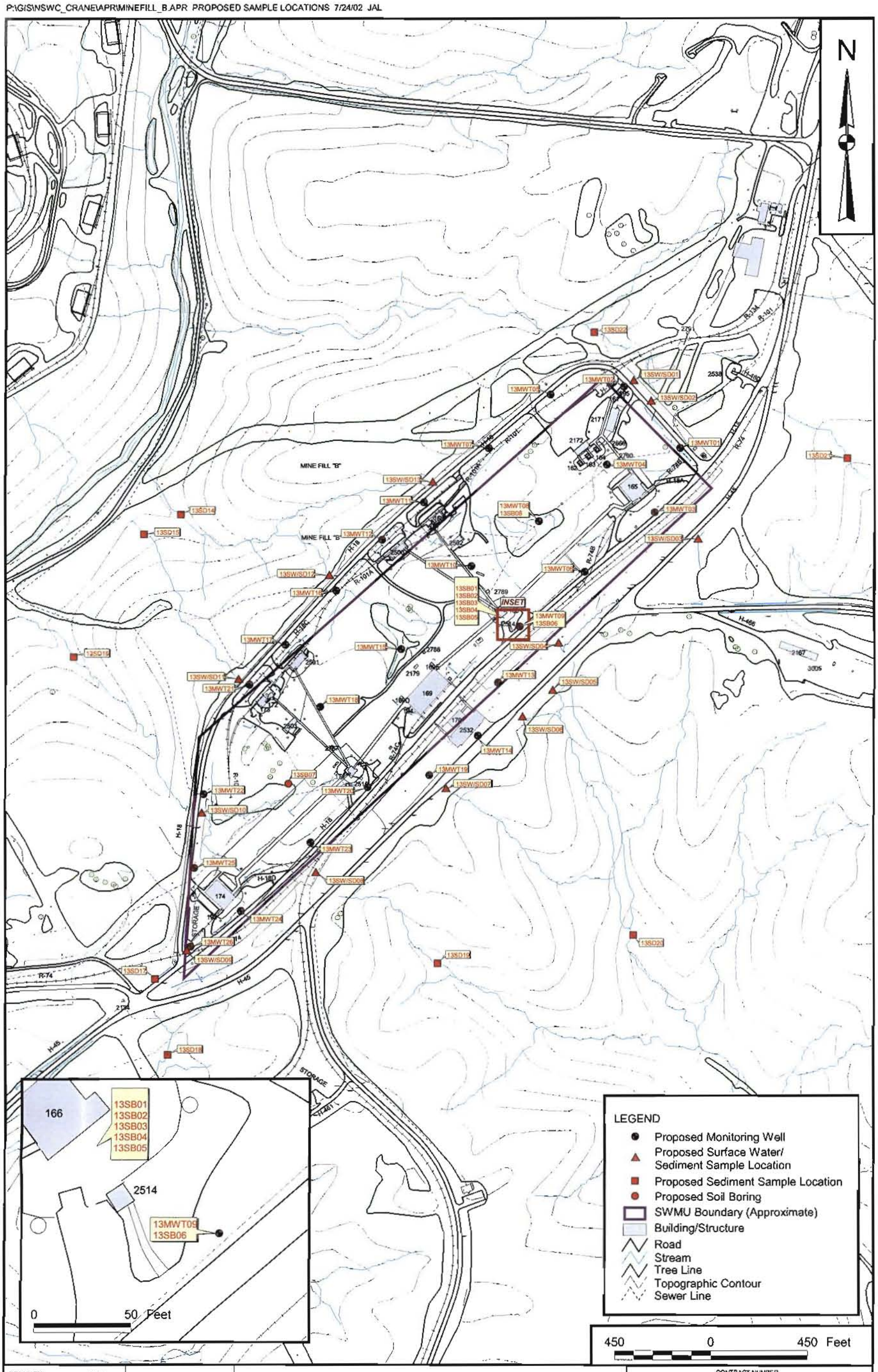

DRAWN BY	DATE
J LAMEY	8/21/01
CHECKED BY	DATE
COST/SCHEDULE-AREA	
SCALE AS NOTED	

PROPOSED SAMPLE LOCATIONS
MINE FILL B (SWMU 13/14)
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

CONTRACT NUMBER	
APPROVED BY	DATE
APPROVED BY	DATE
DRAWING NO. FIGURE 3-2	REV 0

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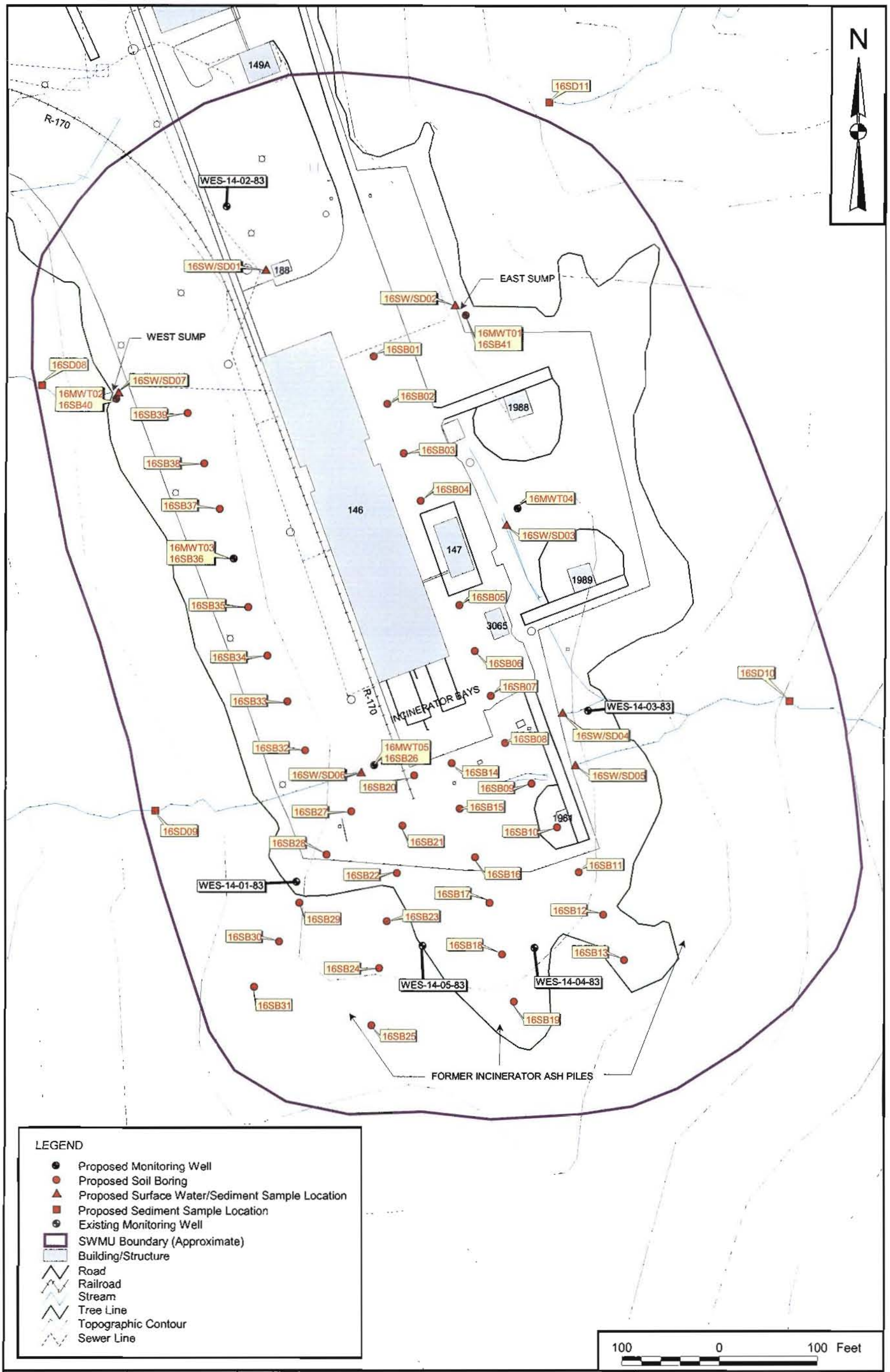

DRAWN BY	DATE			CONTRACT NUMBER
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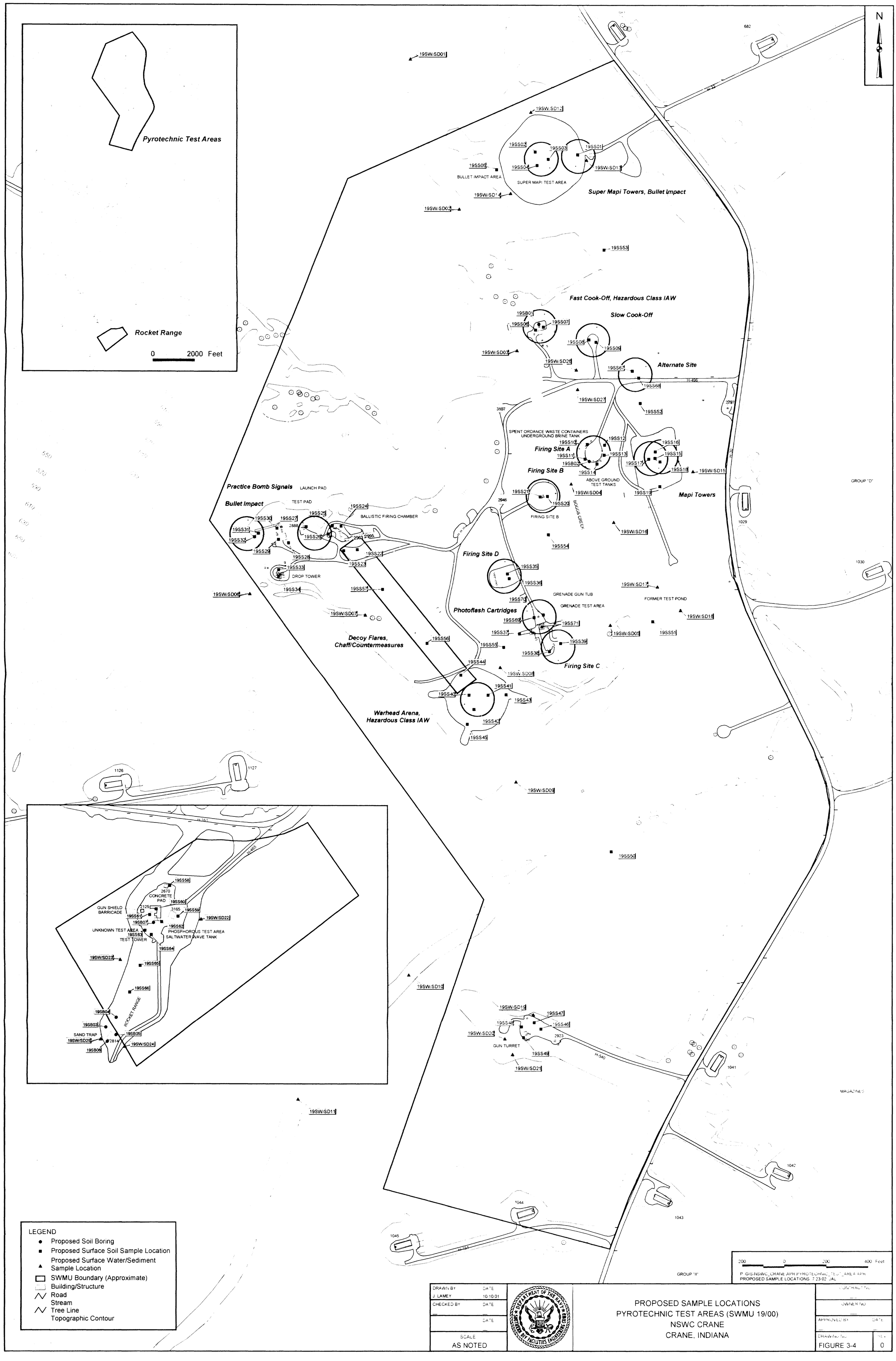
CHECKED BY	DATE			APPROVED BY / DATE
COST/SCHEDULE-AREA				DRAWING NO: FIGURE 3-3 / REV: 0
SCALE: AS NOTED				


Pyrotechnic Test Areas
Rocket Range
0 2000 Feet
N
19SW/SD01
19SW/SD12
19SS02
19SS03
19SS01
19SS05
19SS04
19SW/SD13
BULLET IMPACT AREA
SUPER MAPI TEST AREA
Super Mapi Towers, Bullet Impact
19SW/SD14
19SW/SD02
19SS53
Fast Cook-Off, Hazardous Class IAW
Slow Cook-Off
19SB01
19SS06
19SS07
19SS08
19SS09
19SW/SD03
19SW/SD26
Alternate Site
19SS67
19SS68
19SW/SD27
19SS52
SPENT ORDANCE WASTE CONTAINERS
UNDERGROUND BRINE TANK
19SS10
19SS12
Firing Site A
19SS11
19SS13
19SB02
19SS14
19SS16
19SS15
19SS17
19SS18
19SW/SD15
Firing Site B
ABOVE GROUND
TEST TANKS
19SS21
19SS20
19SW/SD04
19SS19
Mapi Towers
FIRING SITE B
BOGGS CREEK
19SW/SD16
19SS54
Practice Bomb Signals
LAUNCH PAD
Bullet Impact
TEST PAD
19SS30
19SS27
19SS25
19SS24
BALLISTIC FIRING CHAMBER
19SS31
19SS32
19SS26
19SS29
19SS28
19SS22
19SS23
19SS33
DROP TOWER
19SS34
19SS57
19SW/SD06
Firing Site D
19SS35
19SS36
19SW/SD17
GRENADE GUN TUB
GRENADE TEST AREA
FORMER TEST POND
19SS70
Photoflash Cartridges
19SS69
19SS71
19SW/SD18
19SW/SD07
19SS37
19SW/SD05
19SS51
Decoy Flares,
Chaff/Countermeasures
19SS56
19SS55
19SS39
19SS38
Firing Site C
19SS44
19SW/SD08
19SS41
19SS40
19SS43
Warhead Arena,
Hazardous Class IAW
19SS42
19SS45
19SW/SD09
19SS50
19SW/SD10
19SW/SD19
19SS47
19SS48
19SS46
19SW/SD20
GUN TURRET
19SS49
19SW/SD21
19SW/SD11
GROUP "D"
GROUP "II"
MAGAZINES
LEGEND
Proposed Soil Boring
Proposed Surface Soil Sample Location
Proposed Surface Water/Sediment Sample Location
SWMU Boundary (Approximate)
Building/Structure
Road
Stream
Tree Line
Topographic Contour
200 0 200 400 Feet
P:\GIS\NSWC_CRANE\APR\PYROTECHNIC_TEST_AREA.APR
PROPOSED SAMPLE LOCATIONS 7 23 02 JAL
DRAWN BY J. LAMEY DATE 10/10/01
CHECKED BY
SCALE AS NOTED
DEPARTMENT OF THE NAVY SOUTHERN DIV FACILITIES ENGINEERING COMD
PROPOSED SAMPLE LOCATIONS
PYROTECHNIC TEST AREAS (SWMU 19/00)
NSWC CRANE
CRANE, INDIANA
CONTRACT NO
OWNER NO
APPROVED BY
DATE
DRAWING NO FIGURE 3-4
REV 0

4.0 CUSTODY PROCEDURES

Documented sample custody is one of several factors necessary for the admissibility of environmental data as evidence in a court of law. Custody procedures help to satisfy the two major requirements for admissibility: relevance and authenticity. Sample custody is addressed in three parts: field sample collection, laboratory analysis, and final evidence files. Final evidence files, including all original laboratory reports and purge files, are maintained under document control in a secure area. A sample or evidence file is under custody when any one of the following conditions is satisfied:

- The item is in the actual physical possession of an authorized person
- The item is in view of the person after being in his or her possession
- The item was placed in a secure area to prevent tampering
- The item is in a designated and identified secure area with access restricted to authorized personnel only

The Chain-of-Custody (COC) Form is a multi-part, standardized form used to summarize and document pertinent sample information such as sample identification and type, sample matrix, date and time of collection, preservation, and requested analyses. Furthermore, through the sequential signatures of various sample custodians (e.g., sampler, airbill number, laboratory sample custodian), the COC Form documents sample custody and tracking. Laboratory custody procedures will ensure that sample integrity is not compromised from the time of receipt at the laboratory until final data are reported to TtNUS. This requires that the laboratory control all sample handling and storage conditions and circumstances. Custody procedures apply to all environmental and associated field QC samples obtained as part of the data collection system.

4.1 FIELD CUSTODY PROCEDURES

The FOL (or designee) is responsible for the care and custody of the samples collected until they are relinquished to the laboratory or entrusted to a commercial courier. Together, field logbooks and sample documentation including COC Forms provide a record that should allow a technically qualified individual to reconstruct significant field activities without resorting to memory. COC Forms are completed to the fullest extent possible for each sample cooler used for shipment. The forms are legibly completed with waterproof ink and are signed (and dated) by the sampler. COC Forms will include the following information: project name, sample number, time collected, matrix, designated analyses, type of sample, preservative, and name of sampler. Pertinent notes or comments, such as positive results during sample

screening, are also indicated on the COC Form. An example COC Form is included in SOP CTO 166-3 (Appendix H).

Information similar to that contained on the COC Form is provided on the sample label, which is securely attached to the sample bottle. In addition, sample tags will be affixed to the sample bottles and returned by the analytical laboratory for inclusion in the final evidence file. Sample labels and tags will include, at a minimum, the following information: sample number, date and time of collection, analysis required for the sample aliquot in the associated sample container, and a space for the laboratory sample number. The procedures for sample numbering by TtNUS are described in SOP CTO 166-2 (Appendix H), and the procedures for sample numbering by Laucks Testing Laboratories, Inc. and Triangle Laboratories Inc. are described in SOPs LTL-4002 and Triangle-5.01 (Appendix I).

Site conditions during sampling and the care with which samples are handled may factor into the degree to which samples represent the media from which they are collected. This, in turn, could affect the ability of decision makers to make accurate and timely decisions concerning the contamination status of the site. As appropriate, logbooks are assigned to, and maintained by, key field team personnel. The logbooks are used to record daily conditions and activities such as weather conditions, dates and times of significant events, level of PPE used, boring activities, actual sample collection locations, photographs taken, problems encountered during field activities, chemical screening results, and corrective actions taken to overcome problems. In addition, the names of site visitors and the purposes of their visits will be recorded. Field logbook assignments will be recorded in the site logbook or other central file whose location is known by the FOL and the TOM. All field logbook assignments, use, control, and archiving are governed by SOP CTO 166-3 (Appendix H). Examples of all forms to be used during sampling activities are also provided in SOP CTO 166-3 (Appendix H). The FOL is responsible for the maintenance and security of all field records at the end of each workday during field activities. At the completion of field activities, the FOL will forward all field records to the TtNUS TOM. All sample records are eventually docketed into the final evidence file.

SOPs CTO 166-5, CTO 166-6, CTO 166-8, CTO 166-19, and CTO 166-20 (Appendix H) describe procedures for sample screening, packaging, and shipment. A temperature blank will be included in each cooler containing samples for use by the laboratory upon receipt. Each cooler that contains samples to be analyzed for volatile organic compounds will also include a trip blank. Each cooler will be taped shut with strapping tape in at least two places to prevent tampering. Custody seals will be attached as described in SOP CTO 166-3 so that the seals must be broken to open the cooler. Shipment will be made by a public courier at the next scheduled pickup following completion of sample collection.

The following procedures will be used when transferring custody of samples. As previously noted, individual custody records will accompany each sample cooler. The methods of shipment, courier name, and other pertinent information will be entered in the remarks section of the custody record. When transferring samples, the individuals relinquishing and receiving the samples will sign, date, and note the time on the COC Form. The original record (top copy of the multi-part form) will accompany the shipment and the field sampler will retain a copy. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common courier). After COC Forms have been placed within sealed shipping coolers, the signed courier airbills will serve to document COC. Upon arrival at the laboratory, internal laboratory sample custody procedures will be followed (see Section 4.2).

4.2 LABORATORY CUSTODY PROCEDURES

When Laucks Testing Laboratories, Inc., Triangle Laboratories, Inc., and the NSWC Crane Lab receive a shipment of samples, each laboratory's sample custodian will verify that the correct number of coolers has been received. The custodian will examine each cooler's custody seals to verify that they are intact and that the integrity of the environmental samples has been maintained. The custodian will then open each cooler and measure its internal temperature by measuring the temperature of the temperature blank. The temperature reading will be documented in the comments column of the COC Form. In addition, Laucks Testing Laboratories, Inc. will record the temperature reading on the Supplemental Sample Receipt Log, Appendix I, SOP LTL-4002. As presented in the NSWC Crane Lab SOPs CR4052-PD-2500 and CR4052-PD-003, located in Appendix I, the samples will be inspected prior to acceptance into their laboratory. The sample custodian will then sign the COC Form and examine the contents of the cooler. Identification of broken sample containers or discrepancies between the COC Form and sample labels will be recorded. The laboratory will retain the original field COC Forms, providing copies of the forms with the final data package deliverable. All problems or discrepancies noted during this process will be promptly reported to the TtNUS TOM. Samples will be logged into the laboratory information management system. Other pertinent issues relating to laboratory sample custody and tracking are presented in the following laboratory SOPs (Appendix I):

SOP LTL-1002	Document Tracking and Control
SOP LTL-1003	Chain-of-Custody and Documentation Procedures
SOP LTL-1007	Use of Instrument Records and Logbooks
SOP LTL-1013	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
SOP LTL-1019	Controlling, Maintaining, and Monitoring Laboratory Logbooks

SOP LTL-4002	Electronic Sample Entry and Log-In
Triangle 5.01	Sample Receipt, Identification, Handling and Storage
CR4052-PD-003	Internal Sample Chain of Custody

4.3 FINAL EVIDENCE FILES

The Administrative Record at NSWC Crane will be the repository for all documents that constitute evidence relevant to sampling and analysis activities as described in this QAPP. NSWC Crane will be the custodian of the evidence file and will maintain the contents of these files, including all relevant records, reports, logs, field notebooks, pictures, subcontractor reports, and data reviews in a secure, limited-access location and under custody of the NSWC Crane site manager. The control file will include at a minimum:

- Field logbooks
- Field data and data deliverables
- Photographs and negatives
- Drawings
- Soil boring logs
- Laboratory data deliverables (including raw data and calibrations)
- Data validation reports
- Data assessment reports
- Progress reports, QA reports, interim project reports, etc.
- All custody documentation (tags, forms, airbills, etc.)

Upon completion of the contract, all files associated with this investigation will be maintained in the Administrative Record at NSWC Crane and will be available for inspection by the regulatory agencies for at least 50 years. Prior to disposal of all Administrative Records, the records will be offered to U.S. EPA.

5.0 CALIBRATION PROCEDURES AND FREQUENCY

All instrumentation used to perform chemical measurements must be properly calibrated prior to use in order to obtain valid and usable results. Instruments used in the field and in the laboratory will be calibrated in accordance with the procedures governing the use of the instruments. Field SOPs are included in Appendix H, and laboratory SOPs are included in Appendix I. For this investigation, field instrument calibration is described in Section 5.1, and laboratory instrument calibration is outlined in Section 5.2.

5.1 FIELD INSTRUMENT CALIBRATION

Various instruments will be required for field measurements during this investigation and will include the following:

- Multi-parameter water-quality meter
- LaMotte turbidity meter
- Electronic water-level meter
- PID
- HACH DR-800 colorimeter (or equivalent)

Instruments used in the field will be calibrated in accordance with the procedures outlined in specific SOPs (water-quality meter and PID) and the manufacturer's calibration procedures. Calibration of each instrument will be documented on a separate Equipment Calibration Log Form (in SOPs CTO 166-06 and 166-14 of Appendix H). During calibration, an appropriate maintenance check will be performed on each piece of equipment. If damaged or defective parts are identified during the maintenance check and it is determined that the damage could have a negative impact on the instrument's performance, the instrument will be removed from service until the defective parts are repaired or replaced. If the instrument cannot be repaired, a replacement will be procured from the supplier.

Calibration of the HACH DR-800 colorimeter (or equivalent) will be accomplished through accuracy checks performed for each required parameter. Accuracy checks include one or more of standard additions, standard solutions, and reagent blank measurements. The purpose of accuracy checks is to check the performance of the reagents, the colorimeter, and the analytical procedure. Information on the type of accuracy check, its associated parameter, and the QC associated with the accuracy check is

where possible, and appropriate pedigree documentation will be obtained from the supplier. In cases where documentation is not available, the laboratory will analyze the standard and compare the results to a U.S. EPA-known or previous NIST-traceable standard.

Calibrations and associated documentation are required for all laboratory instruments. The documentation for calibrations performed in-house will identify the person performing the calibration, the instrument being calibrated, the standards used for calibration and their concentration values or other pertinent calibration values, the source of the calibration standards, and the date of calibration. Certain instruments (e.g., balances) may be calibrated by a third party. In those cases, the details of calibration as described above and a certification of acceptable performance will be obtained from the third party. The period during which the calibration is valid may appear in the calibration record or may be governed by the SOP.

Calibration procedures, frequency requirements, acceptance criteria, and conditions that require recalibration are described for each analytical procedure in the applicable laboratory SOPs included in Appendix I.

6.0 ANALYTICAL AND MEASUREMENT PROCEDURES

Field measurements and laboratory analytical procedures are presented in this section.

6.1 FIELD MEASUREMENT PROCEDURES

Chemical and physical parameters to be measured using field instrumentation include pH, specific conductance, temperature, turbidity, nitrate, dissolved oxygen, groundwater flow rate, ORP, grain size, sample depth, sample location, and water-level elevation. The field target parameters and the rationales for including them in the analysis scheme for SWMUs 12 and 13, 16, and 19 are presented in Tables 1-3, 1-4, and 1-5, respectively. Measurement of field parameters is described in detail in Section 3. Calibration of field instruments is discussed in Section 5 and in individual field measurement SOPs (Appendix H).

6.2 LABORATORY ANALYTICAL AND MEASUREMENT PROCEDURES

The laboratory analytical procedures (with the exception of the dioxins and furans and dyes) will be performed by Laucks Testing Laboratories, Inc., 940 South Harney Street, Seattle, Washington 98108; (206) 767-5063; FAX (206) 767-5063. Analysis for dioxins and furans will be subcontracted by Laucks to Triangle Laboratories, Inc., 801 Capitola Drive, Durham, NC 27713; (919) 544-5729; FAX (919) 544-5491. Dye analysis will be performed by NSWC Crane Lab, 300 Highway 361, Code 4052, Building 2707, Crane, Indiana, 47522; (812) 854-5506; FAX (812) 854-5054. Both Laucks and Triangle have successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Services Command (NFESC) QA Program and described in the Navy Installation Restoration Laboratory Quality Assurance Guide (NFESC, February 1996). Table 6-1 provides a summary of the laboratory analytical methods and associated laboratory SOPs to be used during this investigation. Laboratory SOPs are included directly or by reference in Appendix I of this QAPP. SOPs included by reference have been distributed previously to recipients of the QAPP.

6.2.1 <u>List of Project Target Compounds and Detection Limits</u>

A list of the laboratory target analytes, project-specific risk-based target levels, and laboratory-specific threshold values (TVs) for organic compounds and miscellaneous parameters, instrument detection limits (IDLs) for metals, and reporting limits (RLs) is provided in Table 1-7. The TVs are defined as the lowest value at which the laboratory can reliably detect and confirm the presence of target compounds. The TVs are based on laboratory experience and MDLs that have been experimentally determined using the

procedures described in Section 6.3 of Laucks Testing Laboratories, Inc., SOP LTL-1011 (which is included in Appendix I of this QAPP). These procedures are based on the method provided in 40 CFR Part 136 Appendix B (FR, 1984). The IDLs provided for metals have been experimentally determined as described in Section 6.2.6 of Laucks Testing Laboratories, Inc., SOP LTL-1011. This procedure is based on the procedure for IDL determination as specified in the U.S. EPA Contract Laboratory Program (CLP) (U.S. EPA, 1995). All environmental data will be reported to the analyte's laboratory-specific and matrix-specific RL, TV, or IDL. An analyte's RL is based on the associated MDL/IDL with adjustments made to ensure that the precision and accuracy requirements of the method are attainable. RLs will be adjusted on a sample-by-sample basis, as necessary, based on dilutions, sample volume, and, for soil samples, percent moisture.

6.2.2 List of Associated Quality Control Samples

Field and laboratory QC samples to be analyzed in support of this project are identified in Section 3.0 and Section 7.0. The analytical SOPs included in Appendix I of this QAPP address minimum QC requirements for each associated analytical method. The SOPs include calibration QC requirements. Details on QC sample usage are provided in Section 7.0.

TABLE 6-1

SUMMARY OF ORGANIC, INORGANIC, AND MISCELLANEOUS ANALYTICAL PROCEDURES FOR SOLID AND AQUEOUS SAMPLES NSWC CRANE, CRANE, INDIANA PAGE 1 OF 3

Analytical Parameter [1]	Preparation Method [2,3,4]	Analytical Method	Preparation/Analytical SOP(s) [5]
EXPLOSIVES			
Explosives SW-846 8330	Aqueous SW-846 8330 [3] Solid SW-846 8330	SW-846 8330	Aqueous LTL-3077 / LTL-8330 Solid LTL-3161 / LTL-8330
Picric Acid/Picramic Acid	Aqueous SW-846 3510C Solid SW-846 3550B	SW-846 8330 Modified	Aqueous LTL-3550/ LTL-8303 Solid LTL-3555/ LTL-8303
METALS			
Metals (except Antimony, Tin, and Mercury	Aqueous SW-846 Methods 3010A (**no** HCl) or 3015 (**no** HCl) Solid SW-846 Method 3050B (include H_2O_2 but **no** HCl)	SW-846 Method 6020	Aqueous LTL-7009 or LTL-7010/ LTL-7202 Solid LTL-7015/ LTL-7202
Antimony and Tin	Aqueous SW-846 Methods 3010A or 3015 (**no** H_2O_2) Solid SW-846 Method 3050B (include HCl but **no** H_2O_2)	SW-846 Method 6020	Aqueous LTL-7009 or LTL-7010/ LTL-7202 Solid LTL-7015/ LTL-7202
Mercury	Aqueous SW-846 Method 7470A Solid SW-846 Method 7471A	Aqueous SW-846 Method 7470A Solid SW-846 Method 7471A	Aqueous LTL-7501 Solid LTL-7501

TABLE 6-1

SUMMARY OF ORGANIC, INORGANIC, AND MISCELLANEOUS ANALYTICAL PROCEDURES FOR SOLID AND AQUEOUS SAMPLES NSWC CRANE, CRANE, INDIANA PAGE 2 OF 3

Analytical Parameter [1]	Preparation Method [2,3,4]	Analytical Method	Preparation/Analytical SOP(s) [5]
VOLATILES			
VOCs SW-846 8260B	Aqueous SW-846 Method 8260B (25 mL purge) Solid SW-846 Method 5035 (5 g purge)	Aqueous SW-846 Method 8260B (25 mL purge) Solid SW-846 Method 8260B (5 g purge)	Aqueous LTL-8265 (low level) Solid LTL-8265
VOCs SW-846 8015B	Aqueous SW-846 Method 8015B (25 mL purge) Solid SW-846 Method 5035 (5 g purge)	SW-846 Method 8015B	LTL-8019
SEMIVOLATILES			
SVOC SW-846 8270C	Aqueous SW-846 Method 3520C Solid SW-846 Method 3550B	SW-846 Method 8270C	Aqueous LTL-3000 / LTL-8276 Solid LTL-3100 / LTL-8276
Selective Ion Monitoring SW-846 Methods 8270C	Aqueous SW-846 Method 3520C Solid SW-846 Method 3550B	SW-846 Methods 8270C-SIM	Aqueous LTL-3001 / LTL-8277 Solid LTL-3450 / LTL-8277
Hexachlorophene and Pentachlorophenol	SW-846 Method 8151A	SW-846 Method 8151A	Aqueous LTL-3510 / LTL-8151 Solid LTL-3011 / LTL-8151
PCBs			
PCBs SW-846 8082	SW-846 Method 8082	SW-846 Method 8082	Aqueous LTL-3202 / LTL-8084 Solid LTL-3302 / LTL-8084

TABLE 6-1

SUMMARY OF ORGANIC, INORGANIC, AND MISCELLANEOUS ANALYTICAL PROCEDURES FOR SOLID AND AQUEOUS SAMPLES NSWC CRANE, CRANE, INDIANA PAGE 3 OF 3

Analytical Parameter [1]	Preparation Method [2,3,4]	Analytical Method	Preparation/Analytical SOP(s) [5]
DIOXINS/FURANS			
Polychlorinated Dibenzodioxins and Dibenzofurans	SW-846 Method 8290	SW-846 Method 8290	Aqueous DSP-161/DHR-182 Solid DSP-105/DHR-182
DYES			
Dyes, NSWC Crane	CR4052-PD-2500	CR4052-PD-2500	CR4052-PD-2500
MISCELLANEOUS PARAMETERS			
Total Organic Carbon	Aqueous SW-846 9060 Solid Lloyd Kahn	Aqueous SW-846 9060 Solid Lloyd Kahn	Aqueous LTL-9115 Solid LTL-9116
Perchlorate	EPA Method 314.0	EPA Method 314.0	Aqueous LTL-9136 Solid LTL-9136
Hardness	Standard Method 2340B	Standard Method 2340B	Calculation
pH	SW-846 Method 9045C	SW-846 Method 9045C	Solid LTL-9113
Cation Exchange Capacity	SW-846 Method 9081	SW-846 Method 9081	Solid LTL-6006
Cyanide	SW-846 Method 9012A	SW-846 Method 9012A	Aqueous and Solid LTL-9104
AVS/SEM[6]	EPA Draft Analytical Method, December 1991	EPA Draft Analytical Method, December 1991	Solid LTL-6008
Nitrate + Nitrite as Nitrate	EPA 353.2	EPA 353.2	Aqueous LTL-9125
Ammonia	EPA 350.1	EPA 350.1	Aqueous and Solid LTL-9109

1 Reference Table 1-7 of Section 1 for the specific compounds.
2 U.S. EPA, 1986. Test methods for Evaluating Solid Wastes, Physical/Chemical Methods. SW-846, Third Edition and subsequent updates.
3 U.S. EPA Chemical Analyses of Water and Wastewater, March 1983.
4 Standard Method for the Analysis of Waters and Wastewaters, American Public Health Association and others, 20th edition, 1998.
5 Laboratory SOPs are included in Appendix I of this QAPP.
6 Laucks is currently developing methodology for these parameters.

7.0 INTERNAL QUALITY CONTROL CHECKS

Field and laboratory QC samples will be analyzed routinely to evaluate overall data quality. This section provides information regarding those internal QC checks. The overall QA objective for this project is to develop and implement procedures for field sampling, chain-of-custody, laboratory analysis, data management, and reporting that will yield results sufficient to support the attainment of the project objectives specified in Section 1.0. Intended data uses, including the list of project target parameters, are described in Section 1.0 of this QAPP. The manner in which decision making will be based on data comparisons is described in Sections 1.4.4 and 8.0. Specific procedures for sampling, chain-of-custody, laboratory instrument calibration, laboratory analysis, internal QC, reporting of data, audits, preventive maintenance of field and laboratory equipment, data management, corrective action, and reporting to management are described in the remaining sections of this QAPP. As part of those evaluations, statistical parameters such as data set variances will be computed that provide direct insight into the variability of target analyte data in soils and ground water.

The PARCC (precision, accuracy, representativeness, comparability, and completeness) parameters are qualitative and quantitative statements regarding the quality characteristics of the data used to support project objectives and, ultimately, environmental decisions. These parameters are presented in the remainder of this section. Equations used to compute accuracy, precision, and completeness values are provided in Section 8.0.

7.1 PRECISION

7.1.1 Definition

Precision is a measure of the degree to which two or more measurements are in agreement and describes the reproducibility of measurements of the same parameter for samples analyzed under similar conditions. A fundamental tenet of using precision measurements for QC is that precision will be bounded by known limits. Results outside these predetermined limits trigger corrective actions.

By definition, chemical solutions are uniform in composition. Therefore, discounting any imprecision caused by the sample matrix, the variability of analytical results for water samples should be relatively low unless suspended material or sample handling and storage introduce additional imprecision. Precision acceptance criteria for aqueous and soil and sediment duplicate samples have been assigned accordingly in Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15, and 7-17. Results outside these limits will trigger corrective actions, which are presented in Table 3-1.

Because of the inherent and unknown heterogeneity of soil samples, the precision of soil field duplicate samples will not be used for QC but will be compared to laboratory precision estimates to gain a perspective on the natural heterogeneity of the soil.

7.1.2 Field Precision Objectives

Field precision is assessed by collecting and measuring field duplicates at a rate of one duplicate per 10 environmental samples. Acceptance limits for field duplicate precision are provided in Table 3-1. This precision estimate encompasses the combined uncertainty associated with sample collection, homogenization, splitting, handling, laboratory and field storage (as applicable), preparation for analysis, and analysis. In contrast, precision estimates obtained from analyzing duplicate laboratory samples incorporate only homogenization, subsampling, preparation for analysis, laboratory storage (as applicable), and analysis uncertainties. Consequently, the field precision estimates [i.e., relative percent difference (RPD) values] should equal or exceed the laboratory precision estimates, on average, for each analyte. If field duplicate precision is significantly different from laboratory duplicate precision, the underlying cause will be investigated to determine whether the observed difference could be artifacts of sampling and analysis. Considerations given to this effort include

- The scale of subsampling for laboratory precision estimates relative to the scale of field duplicate sample size.
- Analytical measurement precision.
- Precision for repeat analysis of the same solid laboratory control sample (LCS).
- Estimated environmental sample grain size relative to LCS grain size.
- Potential natural soil heterogeneity.
- Concentration level of the analyte.

7.1.3 Laboratory Precision Objectives

Laboratory precision QC samples [i.e., laboratory duplicates for inorganic chemicals and matrix spike duplicates (MSDs) for organic chemicals] will be analyzed with a minimum frequency of five percent (i.e., one QC sample per 20 environmental samples). Laboratory precision is measured by comparing RPD values with precision control limits specified in Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15, and 7-17.

7.2 ACCURACY

7.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value. This parameter is assessed by measuring spiked samples [e.g., surrogate spikes or matrix spikes (MSs)] or well-characterized samples of certified analyte concentrations (e.g., LCSs) and by measuring blanks. Accuracy measurements are designed to detect biases resulting from the sample handling and analysis processes. The equations for determining accuracy of an individual MS and a surrogate spike or LCS for this project are provided in Section 8.0. The equations in Section 8.0 do not apply to blank samples, however, because division by zero (the expected amount or added amount) causes the calculated value to be infinite. Instead, acceptance criteria are designed to limit the tolerable amount of contamination while recognizing that non-zero results for blanks are likely, if only because of random error in the measurement process. The laboratory analytical SOPs (Appendix I) limit tolerable blank concentrations. The bias computations for individual MSs, LCSs, and method blanks will be used to control the analysis process by triggering corrective actions as specified in Table 3-1.

7.2.2 Field Accuracy Objectives

Field test kits are operated in accordance with the manufacturer's instructions that accompany the test kit. Accuracy requirements for field measurements are typically ensured through control over the sample collection and handling and through routine instrument calibration. In addition, after field measurements are completed for specific conductance, pH, and turbidity, a check standard is analyzed to verify continued acceptable calibrations. The acceptance criteria for field measurement accuracy are specified in the field SOPs (included in Appendix H). Accuracy of grain size and depositional environment classifications is ensured by requiring that a qualified field person makes those classifications.

Accuracy is also typically monitored through the use of blanks to detect cross-contamination and by monitoring adherence to procedures that prevent sample contamination or degradation. Equipment rinsate blanks will be collected for this investigation to assess cross-contamination via non-disposable sample collection equipment. Ambient condition blanks will not be collected unless site conditions during sampling (e.g., generation of fugitive dust) indicate a need to assess infiltration of airborne contaminants into sampling containers. Source water blanks will be collected to monitor the purity of water used to decontaminate sampling equipment. Accuracy also will be assured qualitatively through adherence to all sample handling, preservation, and holding-time requirements.

7.2.3 Laboratory Accuracy Objectives

Accuracy in the laboratory is measured through the comparison of a spiked sample or LCS result to a known or calculated value and is expressed as a percent recovery (%R). It is also assessed by monitoring the analytical recovery of select surrogate compounds added to samples that are analyzed by organic chromatographic methods. MS and surrogate compound analyses measure the combined accuracy effects of the sample matrix, sample preparation, and sample measurement. LCSs are used to assess the accuracy of laboratory operations with minimal sample matrix effects. Post-digestion spikes (PDSs) are used to assess the accuracy of the analytical measurement on the sample extract or digestate. Each spiked sample will be spiked with representative target analytes for the analysis being performed to ensure that accuracy measures are obtained for each target analyte. Spiking concentrations will equal or approximate the default concentrations detailed in the applicable sample preparation SOPs. LCS and MS analyses are performed at a frequency no less than one per 20 associated samples of like matrix. Laboratory accuracy is assessed via comparison of calculated %R values to accuracy control limits specified in Tables 7-1 through 7-18.

7.3 COMPLETENESS

7.3.1 Definition

Completeness is a measure of the amount of usable, valid analytical data obtained compared to the amount expected to be obtained. Completeness is expressed as a percentage.

7.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid field measurements obtained from all the field measurements taken in the project. Documentation of sample location and depth is planned for each soil and ground water sample. Documentation of grain size and depositional environment is also planned for each soil sample. A completeness criterion of 100 percent applies to these measurements. However, the 100 percent completeness criterion for depositional environment and grain size may be reduced, depending on whether these factors are significant for background comparisons, as determined under the Base-Wide Background Soil Investigation for NSWC Crane. Furthermore, failure to document grain size and depositional environment may be recoverable by inspecting field logs and site maps or through laboratory measurement once the samples are received by the laboratory. Failure to obtain 100 percent of these measurements for field samples will indicate a need for corrective actions designed to recover the missing information. Failure to recover the information will constitute a need to resample, unless the missing data are judged not to adversely affect attainment of project objectives.

Turbidity is a critical parameter that must be determined before aqueous samples are collected to establish attainment of equilibrium, and its completeness criterion is 100%. There are no completeness criteria for dissolved oxygen, nitrate, flow rate, oxidation-reduction potential, water level, pH, specific conductance, and temperature. These are non-critical parameters that are generally determined to verify that appropriate sampling conditions exist prior to sampling or to provide data to evaluate the potential efficacy of monitored natural attenuation (MNA) as a remedial option.

7.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of usable, valid laboratory measurements per matrix obtained for each of the target analytes. Usable, valid results are those that are judged, after data assessment, to represent the sampling populations and to have not been disqualified for use through data validation or data assessment.

Laboratory completeness objectives are 90 percent for each critical target analyte per sample matrix (soil and water). The use of kriging (i.e., geostatistical contouring), which can interpolate missing values, may mitigate some of the adverse effects experienced from loss of data. Furthermore, the impact of missing soil data cannot be quantified in advance of sampling because the impact will depend on which data are missing. The impact of the loss of any other particular datum on attainment of project objectives will be evaluated during data assessment.

Qualifications on the use of data caused by incomplete data sets will be documented in the RFI report.

7.4 REPRESENTATIVENESS

7.4.1 Definition

Representativeness is an expression of the degree to which the data accurately and precisely represent a characteristic of a population or environmental condition existing at the site. Adherence to the sampling and analysis plan (SAP) (Section 3.0) and use of standardized sample collection, handling, preparation, analysis, and reporting procedures ensures that the final data accurately represent the desired populations. Representativeness will be evaluated during data assessment to determine whether each datum belongs to the observed data distribution through outlier testing. The statistical tests to be used are described in Section 8.0. Any anomalies will be investigated to assess their impact on statistical computations.

7.4.2 Measures to Ensure Representativeness of Field Data

Representativeness depends upon the proper design of the sampling program and will be satisfied by ensuring that the SAP (Section 3.0) is followed and that proper sampling techniques are used. Strict adherence to soil type descriptions and care to ensure that a soil sample representing the appropriate depth interval is placed into each sample container will be ensured during sample collection. Well stabilization parameters (See Table 1-6) will be monitored to ensure that ground water wells have attained equilibrium prior to sampling. Upstream surface water and sediment samples will be collected under conditions that are judged to best represent site characteristics in the absence of impacts from the SWMU.

7.4.3 Measures to Ensure Representativeness of Lab Data

Representativeness in the laboratory is ensured or evaluated by using the proper analytical procedures, meeting sample holding times, and analyzing and evaluating field duplicate samples relative to laboratory duplicates. During development of this QAPP, measures to ensure representativeness of the generated data included consideration of past operations, existing analytical data, physical setting, depositional environments, monitoring well placement, spatial coverage of the proposed sampling locations, accessibility to sampling locations, and constraints inherent to the RCRA program. The rationale of the sampling network is presented in detail in Sections 1.0 and 3.0.

7.5 COMPARABILITY

7.5.1 Definition

Comparability is defined as the confidence with which one data set can be compared to another (e.g., between sampling points; between sampling events). Comparability is achieved by using standardized sampling and analysis methods and data reporting formats (including use of consistent units of measure) and by ensuring that reporting and detection limits are sufficiently low to satisfy project detection and quantitation criteria for the duration of the project. The RLs anticipated for this project are presented in Table 1-7. Additionally, consideration was given to seasonal conditions and other environmental variations that could exist to influence analytical results, but no such influences appear to exist for this investigation that would indicate a need to collect samples at times other than those planned for this investigation.

7.5.2 Measures to Ensure Comparability of Field Data

Comparability depends upon the proper design of the sampling program and will be satisfied by ensuring that Section 3.0 of this QAPP is followed and that proper sampling techniques are used. The rationale behind the SAP design is found in Sections 1.0 and 3.0.

7.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented. The use of sampling and analysis methods in this investigation that are comparable in performance to those used in the Base-Wide Background Soil Investigation for NSWC Crane should limit the need to consider biases when making soil data set comparisons for metals. Results will be reported in units that ensure comparability with previous data. The units used for the laboratory measurements are further explained in Section 8.0 of this QAPP.

7.6 LEVEL OF QUALITY CONTROL EFFORT

Several QC samples will be analyzed for this project to provide a means to assess field and laboratory performance. Field QC samples consist of field duplicates, equipment rinsate blanks, source water blanks, temperature blanks, and, at the discretion of the FOL, ambient condition blanks. Each type of field QC sample undergoes the same preservation, analysis, and reporting procedures as the related environmental samples. Frequencies of field QC sample collection and analysis are presented in Table 3-1. The types and numbers of QC samples to be collected in the field are presented in Table 3-3, 3-5, 3-7, and 3-9.

Laboratory QC encompasses a host of other checks performed during sample preparation and analysis. Frequencies for laboratory QC checks are provided in Table 3-1 and in the method-specific laboratory SOPs appended to this QAPP (Appendix I).

7.7 FIELD QUALITY CONTROL CHECKS

TtNUS has established a QC program designed to monitor and assess the quality of field work performed during environmental investigations. That program includes the use of various types of QC samples as indicated in Table 3-1. Some of the samples are identified in Table 3-1 as requiring additional sample material to be collected in the field, even though the actual field QC check is performed in the laboratory.

The field QC samples consist of field duplicates, trip blanks, equipment rinsate blanks, source water blanks, trip blanks, temperature blanks, and, at the discretion of the FOL, ambient condition blanks. Temperature blanks will be included in each cooler submitted to the laboratory to monitor sample storage conditions prior to arrival at the laboratory. With the exception of temperature blanks, each type of field QC sample undergoes the same preservation, analysis, and reporting procedures as the related environmental samples. Target precision and accuracy values, as applicable, for field QC samples are presented in Table 3-1.

7.7.1 Source Water Blanks

Source water blanks are obtained by sampling the analyte-free water and potable water sources used for decontaminating sampling equipment. Source water blanks are used to determine whether the analyte-free water (used for sampling equipment decontamination procedures) or the potable water (used for steam cleaning) may be contributing to sample contamination.

7.7.2 Field Duplicates

Field duplicates will be collected and analyzed for chemical constituents to measure the cumulative uncertainty (i.e., precision) of the sample collection, splitting, handling, storage, preparation, and analysis operations, as well as natural sample heterogeneity that is not eliminated through simple mixing in the field. Field duplicates are two samples prepared by mixing a volume of sample and splitting it into two separate sample containers that are labeled as individual field samples. Solid field duplicates collected for VOC analysis are not mixed in the field. Two samples are taken in rapid succession from separate but closely located positions. For composite samples, field duplicates will be taken after the sample locations are mixed together in order to verify that the soils were sufficiently combined prior to sampling. For ground water and surface water sampling, field duplicates may be generated by collecting individual water samples from the same well or water source in rapid succession rather than splitting a given volume of water. Field duplicates are labeled as individual environmental samples and are not identified to the laboratory as duplicate samples.

7.7.3 Rinsate Blanks

Equipment rinsate blanks or rinsate blanks are obtained under representative field conditions by collecting the rinse water generated by running analyte-free water through sample collection equipment after sampling and decontamination and prior to use. These blanks will be collected to indicate the potential for sample cross-contamination through the use of improperly cleaned sampling equipment.

7.7.4 Trip Blanks

Trip blanks are samples of deionized water that are analyzed for VOCs. These blanks are used to indicate the potential for cross contamination of the samples by VOCs during sample shipment.

7.7.5 Ambient Condition Blanks

Ambient condition blanks are samples of deionized water poured from one container to another in the field to detect the infiltration of airborne contaminants into field samples. These samples will be used at the discretion of the FOL, if the FOL believes that such infiltration is a possibility based on site conditions.

7.7.6 Temperature Blanks

Temperature blanks are vials of water inserted into each sample cooler prior to shipment from the field. The temperature of the temperature blank is measured prior to shipment and upon receipt at the laboratory to assess whether samples were properly cooled during transit.

7.8 LABORATORY QUALITY CONTROL CHECKS

Laucks Testing Laboratories Inc., Triangle Laboratories Inc., and NSWC Crane Lab operate QC programs that ensure the reliability and validity of the analyses performed at the laboratory. Each laboratory's QA plan describes the policies, organization, objectives, QC activities, and specific QA functions used by that laboratory. All analytical procedures are documented as SOPs. Each analytical SOP specifies minimum QC requirements for the procedure. As previously noted, SOPs for all analyses to be performed during this investigation are included in Appendix I of this QAPP. Table 6-1 lists the SOPs associated with each analytical procedure. In addition, the laboratories maintain SOPs regarding general laboratory QA operations. Several of these SOPs, as applicable, are also included in Appendix I. The Table of Contents included in Appendix I lists titles and corresponding numbers for all laboratory SOPs contained in the appendix.

Internal laboratory analytical QC requirements beyond those used for instrument calibration QC are highlighted in the remainder of this section. Additional QC requirements, specific to the NFESC QA Program, are also specified, as applicable, for each of the QC checks. Target precision and accuracy values (control limits) are presented in Tables 7-1 through 7-18. The applicable analytical SOPs should be consulted for calibration QC measures.

7.8.1 Laboratory Control Samples

LCSs provide a means to monitor the overall performance of each step during the analysis, including the sample preparation. These are solid samples (soil and sediment analyses) or blank spikes (water analyses) that contain concentrations of analytes that are known with a specified degree of certainty.

Based on the requirements of the NFESC QA program, LCSs for metals analyses must contain all analytes of interest, whereas LCSs for multiple-analyte organic methods must contain at least two targeted analytes from each major class of compounds subject to analysis. Target analytes for LCSs are listed in Tables 7-2, 7-4, 7-6, 7-8, 7-10, 7-12, 7-14, 7-16, and 7-18.

Based on NFESC QA program requirements, if recovery of an LCS falls outside the control limits (see Tables 7-2, 7-4, 7-6, 7-8, 7-10, 7-12, 7-14, 7-16, and 7-18), the laboratory will reject the data for the analytical batch and take corrective action. The associated samples, extracts, or digestates may be re-analyzed a single time, and, if the LCS recoveries meet acceptance criteria, the data will be reported. If LCS analyte recovery is still outside the acceptance limits, the associated samples in the preparation batch will be reprocessed, if sufficient sample is available and holding times have not lapsed. If re-preparation or re-analysis is not possible, the data will be flagged and the sample delivery group (SDG) narrative will include details of the failed LCS.

7.8.2 Laboratory Duplicates

Laboratory duplicates will be analyzed for metals and miscellaneous parameters to measure the cumulative uncertainty (i.e., precision) of the sample handling, subsampling, preparation, laboratory storage, and analysis operations within the laboratory, as well as sample heterogeneity that is not eliminated through simple mixing in the laboratory. Laboratory duplicates are two subsamples obtained by the laboratory analyst after the sample is mixed. If chemical analysis RPD values exceed QC limits for laboratory duplicates (Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15, and 7-17), the analytical process will be investigated to assess whether the observed RPD is an indication of a deficient analytical system or of excess sample heterogeneity.

7.8.3 Internal Standards

Internal standards are added to each sample analyzed by GC/MS to ensure that the analysis sensitivity and response are stable during every analytical run. Internal standard area counts for samples and blanks must not vary by more than a factor of two (- 50% to + 100%) from the associated 12-hour calibration standard except for dioxin/furan analyses. In the dioxin/furan analyses, the internal standards

are added to the sample prior to extraction. Therefore, dioxin internal standards measure extraction efficiency and must recover within the control limits specified in Table 7-13.

7.8.4 Laboratory Method Blanks

Laboratory method blanks or preparation blanks are an analyte-free matrix prepared and analyzed in accordance with the analytical method employed to determine whether contaminants originating from laboratory sources have been introduced and have affected environmental sample analyses. Analyte-free water is used as a blank for water analyses. A method blank for organic soil sample analysis consists of an aliquot of sand (or in some cases sodium sulfate, Teflon® beads or glass beads) that is subjected to the same preparation and analysis as the environmental samples. The solid method blank results are presented on a dry-weight basis assuming 100 percent solids. Native soils devoid of acid leachable metals do not exist. Therefore, a method blank for inorganic soil sample analysis consists of an aliquot of analyte-free water that is subjected to the same preparation and analysis procedures as the environmental samples undergoing analysis. The aqueous results are normalized to a fictitious soil sample and presented on a dry-weight basis assuming 100 percent solids.

Acceptance criteria for laboratory method blanks and corrective actions for non-compliant results are described in the applicable analytical SOPs included in Appendix I. Under no circumstances should laboratory method blank contaminant values be subtracted from environmental sample analytical results.

7.8.5 Matrix Spikes (MSs)

MSs are environmental samples to which known quantities of analytes are added prior to sample preparation (digestion or extraction). These samples provide information about the effect of the sample matrix on the sample preparation and measurement methodology.

Matrix spikes, to conform to NFESC requirements, will contain as many representative analytes as practicable. For many analyses, the spiking list will consist of most or all of the target analytes. If the MS recovery is not within applicable control limits (as listed in Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15, and 7-17), the laboratory will assess the batch to determine whether the spike results are attributable to a matrix effect or are the result of other problems in the analytical process. Based on NFESC requirements, if all the batch QC elements that are not affected by the sample matrix are in control (e.g., method blank, LCS, calibration checks) and if there is no evidence that spiking was not properly performed, the poor spike recovery may be attributed to matrix effects. In this case, the associated data will be flagged, but repreparation and re-analysis will not be required. If any of the batch QC elements

that are not affected by the sample matrix are out of control or if there is any evidence that spiking may have been improperly performed, the MS sample will be reprocessed through the entire analytical sequence. If insufficient sample is available, or if holding times have passed, the laboratory will flag the associated data. Details of noncompliant and laboratory duplicate results will be included in the SDG narrative.

7.8.6 Matrix Spike Duplicates (MSDs)

MSDs are duplicates of matrix spikes and are used for estimating the precision of organic target analyte analyses. They are used in lieu of simple duplicate samples because native environmental samples frequently do not exhibit detectable levels of organic target analytes, which otherwise prevents the calculation of RPD values. Precision criteria for MSDs are presented in Tables 7-1, 7-5, 7-7, 7-9, 7-11, 7-13, and 7-17. Lab duplicate (inorganics only) criteria are presented in Table 7-3 and 7-15.

7.8.7 Post-Digestion Spikes (PDS)

PDSs are similar to MSs except that the sample digestate, rather than the original soil sample, is spiked. These spikes are analyzed only for metal target analytes if the matrix spike recovery falls outside control limits. Comparing accuracy (%Rs) for PDSs and MSs helps to identify where in the analytical process accuracy problems are occurring. PDSs will contain all target analytes of interest and will be used to assist in determining whether unacceptable MS recoveries are a result of matrix effects.

7.8.8 Surrogates

Surrogates are organic compounds (typically brominated, fluorinated, or isotopically labeled) that are similar in nature to the compounds of concern and are not likely to be present in environmental media. They are spiked into each sample, standard, and method blank before analysis and are used in organic chromatographic analytical procedures as a check of method effectiveness. Corrective actions for noncompliant surrogate recoveries are presented in the relevant SOPs included in Appendix I of this QAPP. Details of noncompliant surrogate recoveries will be included in the SDG narrative. Accuracy criteria for surrogates are included in Tables 7-1, 7-3, 7-5, 7-7, 7-9, 7-11, 7-13, 7-15, and 7-17.

7.8.9 Additional Laboratory QC Checks

Additional internal laboratory QC checks include mass tuning for GC/MS analysis, second-column confirmation for GC and HPLC analyses, etc. Specific QC requirements for each of these QC checks are provided in the applicable SOPs included in Appendix I of this QAPP.

TABLE 7-1

EXPLOSIVE QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
SW-846 METHOD 8330				
1,3,5-Trinitrobenzene	49-130	30	75-115	50
1,3-Dinitrobenzene	50-130	30	75-108	50
2,4,6-Trinitrotoluene	41-146	30	55-141	50
2,4-Dinitrotoluene	51-133	30	81-118	50
2,6-Dinitrotoluene	51-132	30	81-134	50
HMX	48-125	30	64-147	50
2-Nitrotoluene	49-131	30	90-126	50
3-Nitrotoluene	48-133	30	90-130	50
4-Nitrotoluene	47-131	30	92-132	50
4-Amino-2,6-dinitrotoluene	46-140	30	68-139	50
2-Amino-4,6-dinitrotoluene	49-130	30	74-134	50
Tetryl	28-119	30	20-160	50
Nitrobenzene	44-132	30	75-131	50
RDX	45-130	30	75-124	50
1,2-Dinitrobenzene (surrogate)	20-160	NA	20-160	NA
EPA Method 353.2/Modified Army Corps Method				
Nitrocellulose	30-150	50	30-150	50
LAUCKs HPLC Method				
Picric Acid	TBD[2]	TBD	TBD	TBD
Picramic Acid	TBD	TBD	TBD	TBD
4,6-Dinitro-o-cresol (surrogate)	TBD	NA	TBD	NA

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.
2 Limits will be provided by the laboratory prior to the commencement of field activities.

TBD - To be determined
NA - Not applicable

TABLE 7-2

EXPLOSIVE QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
SW-846 METHOD 8330		
1,3,5-Trinitrobenzene	69-120	90-115
1,3-Dinitrobenzene	67-113	95-108
2,4,6-Trinitrotoluene	66-126	83-125
2,4-Dinitrotoluene	70-118	89-113
2,6-Dinitrotoluene	71-117	85-135
HMX	54-115	82-129
2-Nitrotoluene	65-116	93-129
3-Nitrotoluene	63-121	91-133
4-Nitrotoluene	62-118	94-132
4-Amino-2,6-dinitrotoluene	68-117	81-137
2-Amino-4,6-dinitrotoluene	69-118	92-128
Tetryl	55-130	20-160
Nitrobenzene	65-115	97-131
RDX	59-116	90-128
EPA Method 353.2/Modified Army Corps Method		
Nitrocellulose	30-150	30-150
LAUCKs HPLC Method		
Picric Acid	TBD[2]	TBD
Picramic Acid	TBD	TBD

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.
2 Limits will be provided by the laboratory prior to the commencement of field activities.

TBD - To be determined

TABLE 7-3

METALS QUALITY CONTROL LIMITS[1]
MATRIX SPIKE AND LABORATORY DUPLICATE SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
SW-846 METHOD 6020				
Antimony	75-125	20	75-125	20
Arsenic	75-125	20	75-125	20
Barium	75-125	20	75-125	20
Beryllium	75-125	20	75-125	20
Cadmium	75-125	20	75-125	20
Chromium (total)	75-125	20	75-125	20
Cobalt	75-125	20	75-125	20
Copper	75-125	20	75-125	20
Lead	75-125	20	75-125	20
Nickel	75-125	20	75-125	20
Selenium	75-125	20	75-125	20
Silver	75-125	20	75-125	20
Thallium	75-125	20	75-125	20
Tin	75-125	20	75-125	20
Vanadium	75-125	20	75-125	20
Zinc	75-125	20	75-125	20
Aluminum	75-125	20	75-125	20
Calcium	75-125	20	75-125	20
Iron	75-125	20	75-125	20
Magnesium	75-125	20	75-125	20
Manganese	75-125	20	75-125	20
Potassium	75-125	20	75-125	20
Sodium	75-125	20	75-125	20
Titanium	75-125	20	75-125	20
Lithium	75-125	20	75-125	20
Strontium	75-125	20	75-125	20
SW-846 7470A/7471A				
Mercury	75-125	20	75-125	20

1. In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

TABLE 7-4

METALS QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
SW-846 METHOD 6020		
Antimony	80-120	80-120
Arsenic	80-120	80-120
Barium	80-120	80-120
Beryllium	80-120	80-120
Cadmium	80-120	80-120
Chromium (total)	80-120	80-120
Cobalt	80-120	80-120
Copper	80-120	80-120
Lead	80-120	80-120
Nickel	80-120	80-120
Selenium	80-120	80-120
Silver	80-120	80-120
Thallium	80-120	80-120
Tin	80-120	80-120
Vanadium	80-120	80-120
Zinc	80-120	80-120
Aluminum	80-120	80-120
Calcium	80-120	80-120
Iron	80-120	80-120
Magnesium	80-120	80-120
Manganese	80-120	80-120
Potassium	80-120	80-120
Sodium	80-120	80-120
Titanium	80-120	80-120
Lithium	80-120	80-120
Strontium	80-120	80-120
SW-846 7470A/7471A		
Mercury	75-125	75-125

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

TABLE 7-5

VOLATILE ORGANIC COMPOUNDS QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA
PAGE 1 OF 2

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
SW-846 METHOD 8260B				
1,1,1-Trichloroethane	75-125	20	65-135	30
1,1,2,2-Tetrachloroethane	74-125	20	64-135	30
1,1,2-Trichloroethane	75-127	20	65-135	30
1,1-Dichloroethane	72-125	20	62-135	30
1,1-Dichloroethene	59-145	20	65-135	30
1,2-Dichloroethane	68-127	20	58-137	30
1,2-Dichloropropane	70-125	20	60-135	30
2-Butanone	10-125	20	60-135	30
2-Hexanone	70-125	20	60-135	30
4-Methyl-2-pentanone	70-125	20	60-135	30
Acetone	70-125	20	60-135	30
Benzene	62-142	20	65-135	30
Bromodichloromethane	75-125	20	65-135	30
Bromoform	75-125	20	65-135	30
Bromomethane	72-175	20	62-135	30
Carbon disulfide	70-125	20	60-135	30
Carbon tetrachloride	62-125	20	52-135	30
Chlorobenzene	62-135	20	65-135	30
Chloroethane	65-125	20	55-135	30
Chloroform	74-125	20	64-135	30
Chloromethane	75-125	20	65-135	30
cis-1,2-Dichloroethene	75-125	20	65-135	30
cis-1,3-Dichloropropene	74-125	20	64-135	30
Dibromochloromethane	75-125	20	63-135	30
Ethylbenzene	75-125	20	65-135	30
Methylene chloride	75-125	20	65-135	30
Styrene	75-125	20	65-135	30
Tetrachloroethene	71-125	20	61-135	30
Toluene	59-139	20	64-135	30
trans-1,2-Dichloroethene	75-125	20	65-135	30
trans-1,3-Dichloropropene	66-125	20	56-135	30
Trichloroethene	54-141	20	61-135	30
Vinyl chloride	46-134	20	36-144	30

TABLE 7-5

VOLATILE ORGANIC COMPOUNDS QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA
PAGE 2 OF 2

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
Xylenes (Total)	75-125	20	65-135	30
1,2-Dichloroethane-D4 (surrogate)	62-139	NA	52-149	NA
4-Bromofluorobenzene (surrogate)	75-125	NA	65-135	NA
Difluoromethane (surrogate)	60-140	NA	60-140	NA
Toluene-D8 (surrogate)	75-125	NA	65-135	NA
SW-846 METHOD 8015B				
1,4-Dioxane	70-130	20	30-140	50
Acetonitrile	57-126	20	30-140	50
Isobutyl alcohol	63-130	18	30-140	50
Propionitrile	73-124	15	30-140	50
4-Bromofluorobenzene (surrogate)	70-130	NA	50-150	NA

1. In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

NA Not Applicable

TABLE 7-6

VOLATILE ORGANIC COMPOUNDS QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
SW-846 METHOD 8260B		
1,1-Dichloroethene	60-140	60-140
Benzene	60-140	60-140
Chlorobenzene	60-140	60-140
Toluene	60-140	60-140
Trichloroethene	60-140	60-140
SW-846 METHOD 8015B		
1,4-Dioxane	70-130	30-140
Acetonitrile	57-126	30-140
Isobutyl alcohol	63-130	30-140
Propionitrile	73-124	30-140

1. In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

TABLE 7-7

SEMIVOLATILE ORGANIC COMPOUNDS QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA
PAGE 1 OF 2

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
SW-846 METHOD 8270C				
1,2,4-Trichlorobenzene	43-103	20	40-121	30
1,2-Dichlorobenzene	42-155	30	32-135	30
1,3-Dichlorobenzene	36-125	20	26-135	30
1,4-Dichlorobenzene	33-96	20	25-135	30
2,4,5-Trichlorophenol	25-175	20	25-175	30
2,4,6-Trichlorophenol	39-128	20	29-138	30
2,4-Dichlorophenol	46-125	20	36-135	30
2,4-Dimethylphenol	45-139	20	35-149	30
2,4-Dinitrophenol	30-151	20	25-161	30
2-Chloronaphthalene	60-125	20	50-135	30
2-Chlorophenol	41-115	20	40-106	30
2-Methylphenol	25-125	20	25-135	30
2-Nitroaniline	50-125	20	40-135	30
2-Nitrophenol	44-125	20	34-135	30
3,3'-Dichlorobenzidine	29-175	20	25-175	30
3- and 4-Methylphenol	33-125	20	25-135	30
3-Nitroaniline	51-125	20	41-135	30
4,6-Dinitro-2-methylphenol	26-134	20	25-144	30
4-Bromophenyl phenyl ether	53-127	20	43-137	30
4-Chloroaniline	45-136	20	35-146	30
4-Chloro-3-methylphenol	49-121	20	55-120	30
4-Chlorophenyl phenyl ether	51-132	20	41-142	30
4-Nitroaniline	40-143	20	30-153	30
4-Nitrophenol	38-134	20	23-143	37
Bis(2-chloroethoxy)methane	49-125	20	39-135	30
Bis(2-chloroethyl)ether	44-125	20	34-135	30
Bis(2-chloroisopropyl)ether	36-166	20	26-175	30
Bis(2-ethylhexyl)phthalate	33-129	20	25-139	30
Butyl benzyl phthalate	26-125	20	25-135	30
Di-n-butyl phthalate	34-126	20	25-136	30
Di-n-octyl phthalate	38-127	20	28-137	30
Hexachlorobenzene	46-133	20	36-143	30
Hexachlorobutadiene	25-125	20	25-135	30
Hexachlorocyclopentadiene	41-125	20	31-135	30

TABLE 7-7

SEMIVOLATILE ORGANIC COMPOUNDS QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA
PAGE 2 OF 2

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
Hexachloroethane	25-153	20	25-163	30
Isophorone	26-175	20	25-175	30
n-Nitrosodi-n-propylamine	53-128	20	48-118	28
Phenol	33-112	20	41-109	28
2,4,6-Tribromophenol (surrogate)	10-123	NA	35-140	NA
2-Fluorobiphenyl (surrogate)	30-88	NA	35-140	NA
2-Fluorophenol (surrogate)	10-100	NA	35-140	NA
Nitrobenzene-D5 (surrogate)	35-105	NA	35-140	NA
Phenol-D5 (surrogate)	10-94	NA	35-140	NA
Terphenyl-D14 (surrogate)	33-129	NA	35-140	NA
SW-846 METHOD 8270C WITH SIM				
2-Methylnaphthalene	20-160	30	20-160	30
Acenaphthylene	20-160	30	20-160	30
Acenaphthene	20-160	30	20-160	30
Anthracene	20-160	30	20-160	30
Benzo(a)anthracene	20-160	30	20-160	30
Benzo(a)pyrene	20-160	30	20-160	30
Benzo(b)fluoranthene	20-160	30	20-160	30
Benzo(k)fluoranthene	20-160	30	20-160	30
Benzo(g,h,i)perylene	20-160	30	20-160	30
Chrysene	20-160	30	20-160	30
Dibenzo(a,h)anthracene	20-160	30	20-160	30
Fluoranthene	20-160	30	20-160	30
Fluorene	20-160	30	20-160	30
Indeno(1,2,3-c,d)pyrene	20-160	30	20-160	30
Naphthalene	20-160	30	20-160	30
Phenanthrene	20-160	30	20-160	30
Pyrene	20-160	30	20-160	30
1-Fluoronaphthalene (surrogate)	20-160	NA	20-160	NA
Fluorene-D10 (surrogate)	20-160	NA	20-160	NA
Pyrene-D10 (surrogate)	20-160	NA	20-160	NA

1. In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

NA Not Applicable

TABLE 7-8

SEMIVOLATILE ORGANIC COMPOUNDS QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
SW-846 METHOD 8270C		
1,2,4-Trichlorobenzene	22-102	29-114
1,4-Dichlorobenzene	20-197	27-111
2-Chlorophenol	11-117	29-116
4-Chloro-3-methylphenol	40-114	32-120
4-Nitrophenol	10-119	10-147
n-Nitrosodi-n-propylamine	40-123	23-123
Phenol	10-101	24-121
SW-846 METHOD 8270C WITH SIM		
2-Methylnaphthalene	33-93	30-123
Acenaphthylene	19-108	33-113
Acenaphthene	21-98	34-106
Anthracene	34-98	36-113
Benzo(a)anthracene	49-114	39-124
Benzo(a)pyrene	49-109	40-125
Benzo(b)fluoranthene	43-125	37-130
Benzo(k)fluoranthene	44-124	26-134
Benzo(g,h,i)perylene	19-122	17-131
Chrysene	49-112	36-127
Dibenzo(a,h)anthracene	32-109	22-132
Fluoranthene	54-111	43-121
Fluorene	32-102	40-109
Indeno(1,2,3-c,d)pyrene	31-123	25-131
Naphthalene	15-91	31-109
Phenanthrene	31-103	35-104
Pyrene	40-116	34-121

1. In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

TABLE 7-9

PCB QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
SW-846 METHOD 8082				
Aroclor-1260	40-126	30	31-136	50
Decachlorobiphenyl (surrogate)	30-160	NA	20-150	NA
Tetrachloro-m-xylene (surrogate)	25-139	NA	20-150	NA

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

NA Not Applicable

TABLE 7-10

PCB QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
SW-846 METHOD 8082		
Aroclor-1260	39-149	20-160

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

TABLE 7-11

HERBICIDE QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
SW-846 METHOD 8151A				
Hexachlorophene	31-60	50	20-160	50
Pentachlorophenol	20-160	50	20-160	50
2,4-DCPAA (surrogate)	20-160	NA	50-140	NA

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

NA Not Applicable

TABLE 7-12

HERBICIDE QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
SW-846 METHOD 8151A		
Pentachlorophenol	56-99	20-160

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

TABLE 7-13

POLYCHLORINATED DIBENZODIOXIN/FURAN QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA
PAGE 1 OF 2

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
2,3,7,8-TCDD	70-130	20	70-130	20
1,2,3,7,8-PeCDD	70-130	20	70-130	20
1,2,3,6,7,8-HxCDD	70-130	20	70-130	20
1,2,3,4,7,8-HxCDD	70-130	20	70-130	20
1,2,3,7,8,9-HxCDD	70-130	20	70-130	20
1,2,3,4,7,8,9-HpCDD	70-130	20	70-130	20
OCDD	70-130	20	70-130	20
2,3,7,8-TCDF	70-130	20	70-130	20
1,2,3,7,8-PeCDF	70-130	20	70-130	20
2,3,4,7,8-PeCDF	70-130	20	70-130	20
1,2,3,6,7,8-HxCDF	70-130	20	70-130	20
1,2,3,7,8,9-HxCDF	70-130	20	70-130	20
1,2,3,4,7,8-HxCDF	70-130	20	70-130	20
2,3,4,6,7,8-HxCDF	70-130	20	70-130	20
1,2,3,4,6,7,8-HpCDF	70-130	20	70-130	20
1,2,3,4,6,7,8-HpCDF	70-130	20	70-130	20
OCDF	70-130	20	70-130	20
Total TCDD	70-130	20	70-130	20
Total PeCDD	70-130	20	70-130	20
Total HxCDD	70-130	20	70-130	20
Total HpCDD	70-130	20	70-130	20
Total TCDF	70-130	20	70-130	20
Total PeCDF	70-130	20	70-130	20
Total HxCDF	70-130	20	70-130	20
Total HpCDF	70-130	20	70-130	20
INTERNAL STANDARDS				
2,3,7,8-TCDF	40-130	NA	40-130	NA
2,3,7,8-TCDD	40-130	NA	40-130	NA
1,2,3,7,8-PeCDF	40-130	NA	40-130	NA
1,2,3,7,8-PeCDD	40-130	NA	40-130	NA
1,2,3,6,7,8-HxCDF	40-130	NA	40-130	NA
1,2,3,6,7,8-HxCDD	40-130	NA	40-130	NA
1,2,3,4,6,7,8-HpCDF	25-130	NA	25-130	NA
1,2,3,4,6,7,8-HpCDD	25-130	NA	25-130	NA

TABLE 7-13

POLYCHLORINATED DIBENZODIOXIN/FURAN QUALITY CONTROL LIMITS[1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
NSWC CRANE, INDIANA
PAGE 2 OF 2

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
1,2,3,4,6,7,8,9-OCDD	25-130	NA	25-130	NA
SURROGATE STANDARDS				
2,3,4,7,8-PeCDF	40-130	NA	40-130	NA
1,2,3,4,7,8-HxCDF	40-130	NA	40-130	NA
1,2,3,4,7,8-HxCDD	40-130	NA	40-130	NA
1,2,3,4,7,8,9-HpCDF	25-130	NA	25-130	NA
CLEAN UP STANDARDS				
2,3,7,8-TCDD	40-130	NA	40-130	NA
RECOVERY STANDARDS				
1,2,3,4-TCDD	25-400	NA	25-400	NA
1,2,3,7,8,9-HxCDD	25-400	NA	25-400	NA

1 In-house QC limits provided by Triangle Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data is updated.

NA Not Applicable

TABLE 7-14

POLYCHLORINATED DIBENZODIOXIN/FURAN QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
2,3,7,8-TCDD	70-130	20	70-130	20
1,2,3,7,8-PeCDD	70-130	20	70-130	20
1,2,3,6,7,8-HxCDD	70-130	20	70-130	20
1,2,3,4,7,8-HxCDD	70-130	20	70-130	20
1,2,3,7,8,9-HxCDD	70-130	20	70-130	20
1,2,3,4,6,7,8-HpCDF	70-130	20	70-130	20
OCDD	70-130	20	70-130	20
2,3,7,8-TCDF	70-130	20	70-130	20
1,2,3,7,8-PeCDF	70-130	20	70-130	20
2,3,4,7,8-PeCDF	70-130	20	70-130	20
1,2,3,6,7,8-HxCDF	70-130	20	70-130	20
1,2,3,7,8,9-HxCDF	70-130	20	70-130	20
1,2,3,4,7,8-HxCDF	70-130	20	70-130	20
2,3,4,6,7,8-HxCDF	70-130	20	70-130	20
1,2,3,4,6,7,8-HpCDF	70-130	20	70-130	20
1,2,3,4,7,8,9-HpCDF	70-130	20	70-130	20
OCDF	70-130	20	70-130	20
Total TCDD	70-130	20	70-130	20
Total PeCDD	70-130	20	70-130	20
Total HxCDD	70-130	20	70-130	20
Total HpCDD	70-130	20	70-130	20
Total TCDF	70-130	20	70-130	20
Total PeCDF	70-130	20	70-130	20
Total HxCDF	70-130	20	70-130	20
Total HpCDF	70-130	20	70-130	20

1 In-house QC limits provided by Triangle Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data are updated.

TABLE 7-15

MISCELLANEOUS PARAMETERS QUALITY CONTROL LIMITS[1]
MATRIX SPIKE AND LABORATORY DUPLICATE SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix		Solid Matrix	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
Total Organic Carbon 9060/Lloyd Khan	80-120	30	50-150	50
Perchlorate EPA 314	TBD[2]	TBD	TBD	TBD
Cyanide SW-846 9012B	TBD	TBD	TBD	TBD
Nitrate + Nitrite as Nitrate	68-122	10	NA	NA
Ammonia	53-120	10	50-128	30

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data are updated.
2 Limits will be provided by the laboratory prior to the commencement of field activities.

TBD To be determined.

TABLE 7-16

MISCELLANEOUS PARAMETERS QUALITY CONTROL LIMITS[1]
LABORATORY CONTROL SAMPLES
NSWC CRANE, INDIANA

Chemical	Aqueous Matrix	Solid Matrix
	Accuracy (%R)	Accuracy (%R)
Total Organic Carbon 9060/Lloyd Khan	TBD[2]	80-120
Perchlorate EPA 314	TBD	TBD
Cyanide SW-846 9012B	TBD	TBD
Nitrate + Nitrite as Nitrate	90-110	NA
Ammonia	90-110	90-110

1 In-house QC limits provided by Laucks Testing Laboratories, Inc. QC limits may change prior to SWMU investigations as laboratory QC data are updated.
2 Limits will be provided by the laboratory prior to the commencement of field activities.

TBD To be determined.

TABLE 7-17

QUALITY CONTROL LIMITS [1]
MATRIX SPIKE/MATRIX SPIKE DUPLICATE SAMPLES AND SURROGATE SPIKES
DYE ANALYSES
NSWC CRANE, INDIANA

Chemical	Solid Matrix [2]		Aqueous Matrix [3]	
	Accuracy (%R)	Precision (RPD)	Accuracy (%R)	Precision (RPD)
Acid Blue 9	3 – 9	35	16 - 113	37
Acid Blue 45	46 – 75	35	0 - 117	53
Acid Orange 10	191 – 213	35	50 - 114	20
Acid Red 64	61 – 69	35	43 - 109	22
Acid Yellow 23	55 – 74	35	48 - 111	20
Acid Yellow 73	61 – 68	35	47 - 110	20
1-Aminoanthraquinone	73 – 94	35	41 - 96	20
2-Aminoanthraquinone	42 – 46	35	36 - 87	21
Basic Violet 10	93 – 105	35	46 - 111	21
Disperse Blue 14	34 – 73	35	35 - 109	26
Disperse Red 9	40 – 48	35	46 - 115	22
Disperse Violet 1	45 – 68	35	46 - 100	20
Solvent Green 3	25 – 72	35	2 - 84	47
Solvent Orange 3	51 – 54	35	41 - 92	20
Solvent Orange 7	58 – 94	35	37 - 65	20
Solvent Red 1	25 – 88	35	38 - 74	20
Solvent Red 24	50-150	35	34 - 102	25
Solvent Yellow 2	41 - 47	35	47 - 104	20
Solvent Yellow 3	210 - 225	35	49 - 116	20
Solvent Yellow 14	41 - 44	35	42 - 88	20
Solvent Yellow 33	35 - 41	35	46 - 106	20
Anthracene	42 - 57	NA	0 - 106	NA
Benzanthrone	99 - 113	NA	32 - 120	NA

1 In-house limits provided by the NSWC Crane Lab. QC limits may change prior to SWMU investigations as laboratory QC data is updated.
2 Soil QC limits are based on the short gradient because quantitation of soil sample results will be made from the short gradient.
3 Aqueous QC limits are based on the long gradient because quantitation for aqueous samples will be made from the long gradient.
NA - Not Applicable

TABLE 7-18

QUALITY CONTROL LIMITS [1]
LABORATORY CONTROL SAMPLES
DYE ANALYSES
NSWC CRANE, INDIANA

Chemical	Solid Matrix [2]	Aqueous Matrix [3]
	Accuracy (%R)	Accuracy (%R)
Acid Blue 9	3 – 9	16 – 113
Acid Blue 45	46 – 75	0 – 117
Acid Orange 10	191 – 213	50 – 114
Acid Red 64	61 – 69	43 – 109
Acid Yellow 23	55 – 74	48 – 111
Acid Yellow 73	61 – 68	47 – 110
1-Aminoanthraquinone	73 – 94	41 – 96
2-Aminoanthraquinone	42 – 46	36 – 87
Basic Violet 10	93 – 105	46 – 111
Disperse Blue 14	34 – 73	35 – 109
Disperse Red 9	40 – 48	46 – 115
Disperse Violet 1	45 – 68	46 – 100
Solvent Green 3	25 – 72	2 – 84
Solvent Orange 3	51 – 54	41 – 92
Solvent Orange 7	58 – 94	37 – 65
Solvent Red 1	25 – 88	38 – 74
Solvent Red 24	50 – 150	34 – 102
Solvent Yellow 2	41 – 47	47 – 104
Solvent Yellow 3	210 – 225	49 – 116
Solvent Yellow 14	41 – 44	42 – 88
Solvent Yellow 33	35 – 41	46 – 106
Anthracene	42 – 57	0 – 106
Benzanthrone	99 – 113	32 – 120

1 In-house limits provided by the NSWC Crane Lab. QC limits may change prior to SWMU investigations as laboratory QC data is updated.
2 Soil QC limits are based on the short gradient because quantitation of soil sample results will be made from the short gradient.
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8.0 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING

This section describes the procedures to be used for data reduction, validation, assessment, and reporting. Data generated during the course of the field investigations will be maintained in hard-copy form in the Administrative Record at NSWC Crane.

8.1 DATA REDUCTION

8.1.1 Field Data Reduction Procedures

All field logs containing observations will be inspected and approved by the FOL. All field observations will be recorded in the logs immediately after observations are made. No data reduction will be required in the field.

Concentration measurements will be reported in the units indicated in Table 1-6.

If errors are made in recording or transcribing observations, erroneous observations will be legibly crossed out using a single line, initialed, dated by the field member, and corrected in a space adjacent to the crossed-out entry. The FOL is responsible for assuring that errors are identified and assessed relative to the intent of the QAPP.

Errors judged to affect the use of the sample results within the context of this investigation will be brought to the immediate attention of the TOM.

8.1.2 Laboratory Data Reduction Procedures

Data reduction will be completed by Laucks Testing Laboratories, Inc., Triangle Laboratories, and NSWC Crane Lab in accordance with the method-specific laboratory SOPs included in Appendix I. In addition, data are reviewed in accordance with the laboratory QA plans.

Laboratory analytical data will be reported using standard concentration units to ensure comparability with previous analytical results. Ground water and surface water sample results will be reported in units of micrograms per liter (µg/L); soil and sediment sample results will be reported on a dry-weight basis in units of micrograms per kilogram (µg/kg) for organic parameters and milligrams per kilogram (mg/kg) for inorganic parameters. Dioxin results will be reported in units of picograms per liter (pg/L) for aqueous samples and picograms per gram (pg/g) for solid samples.

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8.2 DATA VALIDATION

Validation of field measurements and laboratory analytical data is presented in this section.

8.2.1 Procedures Used to Validate Field Data

Field measurements will not be subjected to a formal data validation process. Validation of field data will be limited to real-time inspection by the FOL of observations relative to actual site conditions and activities. In addition, field technicians will ensure that the equipment used for sample collection is performing adequately via compliance with the applicable SOPs.

8.2.2 Procedures Used to Validate Laboratory Data

One hundred percent of the laboratory analytical data will be subjected to data validation to ensure that the data are of evidentiary quality. Validation of analytical data will be completed by the TtNUS Environmental Chemistry/Toxicology Department, located in the TtNUS Pittsburgh office. Final review and approval of validation deliverables will be completed by the department's data validation manager. The data validation manager is responsible for ensuring that data validation deliverables are prepared in accordance with the guidance methods specified in this QAPP and are complete and correct.

Prior to statistical analyses, analytical results will be validated versus the applicable analytical methods, the SOPs included in Appendix I, and the requirements of this QAPP. Validation of these data will conform to the U.S. EPA Region 5 Standard Operating Procedures for Validation of CLP Inorganic and Organic Data (U.S. EPA, 1995a/b) and the National Functional Guidelines for Inorganic and Organic Data Review (U.S. EPA, 1994a/b) to the greatest extent practicable. Data validators will review the chemical analytical data packages submitted by the laboratory. The data validators will check that the data were obtained using approved methodology, that the appropriate level of QC and reporting was conducted, and that the results are in conformance with QC criteria.

On the basis of the data validation results, the data validator will generate a report describing detected data limitations. The report will be reviewed internally by the data validation manager prior to submittal to the TOM.

Compliance with quantitative QC objectives for laboratory accuracy and precision, as outlined in Tables 7-2 through 7-18, will be evaluated during data validation. In general, data validation requires that data be evaluated batch by batch based on the results of quality indicators for the respective batches.

8.3 DATA ASSESSMENT

The assessment of data obtained from this investigation is a critical part of determining the next step in data collection and decision making. It must be determined whether the data are of appropriate type, quality, quantity, and representativeness to support the project objectives. The effect of the loss of data deemed unacceptable for use, for whatever reason, will be evaluated.

8.3.1 Reconciliation with Data Quality Objectives

Field data will be examined immediately after generation for errors. Laboratory data will be examined upon receipt from the laboratory in a cascading series of evaluations.

After data validation, the data will be reconciled with DQOs to determine whether sufficient data of acceptable quality are available for decision making. In addition to the evaluations described in the following sections, a series of inspections and statistical analyses will be performed to estimate several of the data set characteristics. The statistical evaluations will include simple summary statistics for target analytes, such as the maximum concentration, minimum concentration, number of samples exhibiting no detectable analyte, the number of samples exhibiting detectable analytes, and the proportion of samples with detectable and undetectable analytes. The data will be presented in a tabular format. These inspections and statistical analyses will be designed to

- Identify deviations, if any, from the field sampling SOPs (inspection).
- Identify deviations, if any, from the laboratory analytical SOPs (inspection).
- Identify deviations, if any, from the QAPP (inspection).
- Identify deviations, if any, from the data validation process (inspection).
- Identify and explain the impacts of elevated MDLs, TVs, and IDLs (inspection).
- Identify unusable data (i.e., data qualified as "R") (inspection).
- Evaluate project assumptions such as ground water direction (inspection).

- Characterize data set distributions (e.g., Shapiro-Wilk W test) if enough data are available (statistics).

- Identify unanticipated data set characteristics such as a laboratory variance greater than the sampling variance (i.e., ANOVA, *t*-test) if enough data are available (statistics).

- Identify and evaluate potential data outliers (95% confidence goodness-of-fit test on probability plot data). The plotted data will be transformed, if necessary, depending on the observed distribution (statistics).

- Evaluate adherence to investigation objectives and decision rules (inspection and statistics, as applicable).

- Ensure completion of corrective actions (inspection).

- Evaluate effects of deviations from planned procedures and processes on the interpretation and utility of the data (inspection and statistics, as applicable).

- Identify the existence of remaining data gaps (inspection/statistics).

For statistical comparisons and mathematical manipulations, analytes that are not detected at the applicable sample-specific MDL, IDL, or TV, as appropriate, will be represented by a concentration equal to one-half the sample-specific MDL, IDL, or TV.

Statistical tests for outlier validity will be based on the Procedural Guidance for Statistically Analyzing Environmental Background Data (NFEC, 1998). Potential outliers will be removed if a review of field and laboratory documents indicates that the results are true outliers. If no identifiable reason for the outlier can be identified, the datum will not be removed from the data set.

If necessary, investigation objectives may be revised in anticipation of additional data collection.

The suitability of any given statistical test will be assessed based on the completeness of the data sets and the conditions observed at the site. For example, when a single data value is available for soils or water samples at a given sampling location, statistical tests cannot be conducted for that individual sampling location. However, pooling of data across sampling locations may be possible and, if logical to do so, may be implemented at the discretion of the TOM. For example, when COPCs are evaluated,

multiple soil sample results of a given depth and grain size within a depositional environment may be pooled for statistical comparison to the background data set from soil of the same depth, grain size, and depositional environment. Statistical testing will generally be conducted at the 5 percent significance level. Statistical testing at other significance levels may also be warranted to provide perspective on the results of testing a 5 percent significance. If other significance levels are used, they will be supported with rationale for their use.

8.3.2 Precision Assessment

As presented in Section 7.1.3, laboratory duplicate samples (for inorganic analyses) and MSD samples (for organic analyses) will be prepared and analyzed at a minimum frequency of one per every 20 environmental samples per matrix. As described in Section 7.1.2, field duplicate samples also will be collected at a minimum frequency of one per 10 environmental samples per matrix. The RPD between a sample or MS (Sample 1) and its duplicate or MSD (Sample 2) is calculated for chemical analyses using to the following formula:

$$\text{RPD} = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} \text{ X } 100\,\%$$

If field duplicate precision is significantly different from laboratory duplicate precision, the underlying cause will be investigated to determine whether the observed difference could be artifacts of sampling and analysis. Because of the inherent and unknown heterogeneity of soil samples, the precision of soil field duplicates will not be used for QC but will be used to gain a perspective on the natural heterogeneity of the soil.

8.3.3 Accuracy Assessment

Sample collection accuracy cannot be evaluated because there is no standard by which to judge such accuracy. Instead of a qualitative evaluation of sample collection accuracy, compliance with field SOPs, as described in Appendix H, will be the metric. Background comparisons of data generated by similar sampling and analysis methods incorporate similar biases and are expected to be directly comparable without any adjustments or compensations.

Accuracy of chemical analyses will be assessed through the use of surrogate spikes, MSs, PDSs, LCSs, calibration check standards, internal standards, and blanks. Blanks will be used to infer the potential for positive biases because of contamination. To assure the accuracy of the analytical procedures, at least

one of every 20 environmental samples will be spiked with known amounts of target analytes (i.e., MSs) prior to preparation for analysis. The spiked samples will be analyzed and the concentrations of each target analyte observed in the spiked sample will be compared to the reported value of the analyte in the unspiked sample to determine the %R of the analyte. Control charts are plotted by the laboratory for each target analyte and are kept on matrix- and analyte-specific bases. The %R for a spiked sample is calculated by using the following formula:

$$\%R = \frac{\text{Amount in Spiked Sample} - \text{Amount in Sample}}{\text{Known Amount Added}} \text{ X } 100\,\%$$

As discussed in Section 7.2.3, LCSs and surrogate spikes are also analyzed to assess accuracy. The %R calculation for LCSs and surrogate spikes is as follows:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \text{ X } 100\,\%$$

Accuracy measurements will be used to detect biases resulting from the sample handling and analysis process.

8.3.4 Completeness Assessment

Completeness for this project will be determined based on the number of sample results for each target analyte and each sample type that are usable as determined through data validation and data assessment. Data values rejected during data validation (indicated by an "R" or "UR" flag) will be considered unusable unless additional review and documentation by one or more technical team members demonstrates that the rejection was erroneous. To monitor completeness, the number of usable, valid results for each matrix type and analyte will be counted and compared to the completeness objectives in Sections 7.3.2 and 7.3.3.

Percent completeness will be calculated using the following equation:

$$\%\,\text{Completeness} = \frac{(\text{number of valid measurements})}{(\text{number of measurements planned})} \text{ x } 100\%$$

Because the many parameters to be measured for this project are interrelated in many different ways, a single completeness criterion cannot be established for the project. Instead, the ability to attain project

objectives will be evaluated at the end of each sampling round by how effectively the necessary decisions and data evaluations can be made.

8.3.5 Representativeness

Representativeness of field data will be evaluated by ensuring that the SAP has been followed and that proper sampling techniques were used. Representativeness of laboratory data will be evaluated by ensuring that proper analytical procedures were followed and sample holding times were met and by evaluating field duplicates relative to laboratory duplicates.

8.3.6 Comparability

Comparability will be evaluated by ensuring that standardized sampling and analysis methods and data reporting formats are used and that reporting limits and threshold values are sufficiently low to satisfy project detection and quantitation criteria.

8.4 DATA REPORTING

8.4.1 Field Data Reporting

Field data will be transcribed from the site logbooks or sample logsheets to the electronic database and will be reviewed for accuracy by an independent reviewer.

All records regarding field measurements (i.e., field logbooks, sampling logbooks, and sample logsheets) will be placed in the TtNUS central files upon completion of the field effort. Entry of these results in the database will require removal of these records from the files. Outcards (date, person, subject matter) will be used to document the removal of any such documentation from the files. After database entry is complete, all records will be copied for placement in the TtNUS central files. All original records will be sent to NSWC Crane for inclusion in the final evidence files, as described in Section 4.3.

8.4.2 Laboratory Data Reporting

To achieve the investigation objectives, a confirmational level of analytical quality is needed. This provides the highest level of data quality necessary to address potential risks. These analyses require full documentation of the chosen analytical methods and sample preparation steps, data packages, and data validation sufficient to provide defensible data. QC must be sufficient to define the overall precision and accuracy of these procedures. Therefore, data reported by Laucks, Triangle, and the NSWC Crane

Laboratories for all analytical fractions will be in a CLP-like reporting format. Hard-copy data deliverables will be generated at the time of analysis. All pertinent QC data (including raw data and summary forms for blanks, standards analysis, calibration information, etc.) will be provided for all analyses. Case narratives will be provided for each SDG. SOP LTL-1018 (Appendix I) provides further details regarding the information that will be included in CLP-type packages produced by Laucks.

Validation will be completed using the hard-copy data. After validation, the validated data will be reviewed by the data validation manager and the validation qualifiers will be entered into the electronic database and then subjected to independent review for accuracy. During this review process, the electronic database printout also will be compared with the hard-copy data to ensure that the hard-copy data and electronic data are consistent.

8.4.3 Project Reports

With each round of data collection, data will be summarized and assessed to determine whether additional data collection is required for determining the extent of contamination at SWMUs 12, 13, 16, and 19. When no more data collection is necessary, a Phase III RFI report will be issued that summarizes the findings of the investigation. If contaminants are detected, the location, concentrations, and boundaries of representative contaminants will be described and depicted on summary maps and diagrams, as appropriate. The results of ground water data evaluations, used to estimate ground water contaminant concentrations relative to RBTLs and background values at the study areas, will be summarized. The evaluation of soil data will also be presented that summarize the measured soil concentrations relative to background soil concentrations and RBTLs. Discussions with U. S. EPA Region 5 concerning the need for future investigations will be summarized, and, if appropriate, recommendations for future investigations will be presented.

Recipients of the Phase III RFI report will include EPA Region 5, IDEM, Navy SOUTHDIV, and NSWC Crane.

8.5 DATA ACQUISITION REQUIREMENTS AND DATA MANAGEMENT

The overall data management scheme and specific requirements for database content, format, and integrity are presented in the TtNUS CTO-126 Data Management Plan (Appendix F). A brief summary is presented in the following paragraphs.

Data acquisition and management will begin with the identification and collection of past data and newly acquired project samples. The samples will be labeled and tagged, packaged for shipment, and shipped to the analytical laboratory in accordance with TtNUS SOPs provided in Appendix H. The samples will be received at the laboratory and analyzed, the analytical results will be reported by the analyst along with QC check data, and the data will be reviewed within the laboratory, in accordance with laboratory SOPs provided in Appendix I. Data will then be transmitted from the laboratory in both hard-copy and electronic formats according to laboratory SOPs provided in Appendix I. The data deliverable requirements are specified in TtNUS's basic ordering agreements with analytical laboratories. This agreement requires the analytical laboratory to provide data in both hard-copy and electronic format. Upon receipt by TtNUS, the data will be validated, analyzed, assessed, and ultimately archived. A copy of the data deliverable requirements will be provided to the NSWC Crane Lab prior to the commencement of field activities.

The electronic database will include pertinent sampling information such as sample number, sampling date, and sample point location, as well as analytical information. Sample-specific TVs, MDLs, IDLs, and RLs will be reported for nondetected analytes. Units will be clearly summarized in the database and will conform to those identified in Section 7.0. The original electronic diskettes and data validation reports for this investigation will be maintained in the Administrative Record at NSWC Crane, and copies will be maintained in TtNUS central files.

9.0 PERFORMANCE AND SYSTEM AUDITS

Performance and system audits will be conducted periodically to ensure that work is being implemented in accordance with the approved QAPP and in an overall satisfactory manner. Some examples of pertinent audits are as follows:

- The FOL will supervise and check daily that the field observations are made accurately, equipment is thoroughly decontaminated, samples are collected and handled properly, and fieldwork is documented accurately and neatly.

- The TOM will maintain contact with the FOL and data validation manager to ensure that management of the acquired data proceeds in an organized and expeditious manner.

Details regarding additional audit responsibilities, frequency, and procedures are provided in the remainder of this section. Field performance and system audits are addressed in Section 9.1. Laboratory performance and system audits are addressed in Section 9.2.

9.1 FIELD PERFORMANCE AND SYSTEM AUDITS

This section presents the responsibilities, frequencies, and procedures associated with internal and external field performance and system audits.

9.1.1 Internal Field Audits

9.1.1.1 Internal Field Audit Responsibilities

In addition to the daily checks performed by the FOL, the TtNUS QAM or designee may conduct an independent performance and system audit of field activities. Such audits are scheduled as part of the NSWC Crane RCRA Environmental Investigation Program, which includes this and other environmental projects. Individual projects are selected for audit by the QAM without the involvement of the TOM. If a formal field audit is conducted for this study, the QAM (or designee) will be responsible for ensuring that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being performed in accordance with the approved QAPP and SOPs.

9.1.1.2 Internal Field Audit Frequency

As explained in Section 9.1.1.1, internal field audits are scheduled as part of the NSWC Crane RCRA Environmental Investigation Program, which includes this and other environmental projects. Individual projects are selected for audit by the QAM without the involvement of the TOM.

9.1.1.3 Internal Field Audit Procedure

Internal field audits will be conducted in accordance with the following procedure:

- Prior to an audit, the auditor will prepare a detailed checklist to be used as an auditing guide. An example audit checklist is provided in Appendix G.

- Upon arrival at the audit location, the auditor will conduct a pre-audit meeting with the responsible management of the organization or project to be reviewed.

- Field audits will include a review of required project documentation (logbooks, sample log sheets, etc.) and field operations (sample COC, sample handling, etc.) to evaluate completeness and compliance with applicable SOPs.

- The audit checklist will be used to record observations, including any noted nonconformances.

- A formal post-audit debriefing will be conducted, and potential immediate corrective actions will be discussed.

- The auditor will generate a formal audit report that will address corrective actions. The auditor will provide this report to the TOM.

- The TOM will ensure that all corrective actions are addressed and will provide written verification of corrective action implementation to the auditor.

- The auditor will manage corrective action verification and audit closure.

- The following audit records will be maintained by the QAM:

  - Audit checklists
  - Audit reports
  - Response evaluations
  - Verification of corrective actions
  - Follow-up checklists and audit reports

9.1.2 External Field Audits

9.1.2.1 External Field Audit Responsibilities

IDEM, U.S. EPA Region 5, or both may conduct external field audits.

9.1.2.2 External Field Audit Frequency

External field audits may be conducted at any time during field activities at the discretion of IDEM and U.S. EPA Region 5. If an audit is to be conducted, scheduling should be coordinated through the TtNUS QAM to ensure that personnel and equipment are available as necessary. Personnel being audited may or may not be informed of the impending audit at the discretion and request of the auditing body.

9.1.2.3 Overview of the External Field Audit Process

External audit procedures are at the discretion of U.S. EPA Region 5 and IDEM.

9.2 LABORATORY PERFORMANCE AND SYSTEMS AUDITS

This section presents the responsibilities, frequencies, and procedures associated with internal and external laboratory performance and system audits.

9.2.1 Internal Laboratory Audits

9.2.1.1 Internal Laboratory Audit Responsibilities

The QAM or appropriate designee of the subcontracted laboratory performs routine internal audits of the laboratory. The U.S. Navy, through the NFESC, also conducts internal laboratory audits. TtNUS holds no responsibility for such audits. Performance and system audits of laboratories are coordinated through the NFESC by an independent QA contractor. It is the responsibility of the NFESC and its contractor to

ensure that the subcontracted laboratories comply with good laboratory practices and the general requirements of all analytical services provided by the laboratories.

9.2.1.2 Internal Laboratory Audit Frequency

Internal audits are performed approximately annually at Laucks Testing Laboratories and Triangle Laboratories. In addition, each laboratory department at Laucks Laboratories and Triangle Laboratories analyzes blind performance evaluation (PE) samples as described in their applicable SOPs (Appendix I) and QA plans.

The NSWC Crane Lab also monitors the quality of work as described in the Crane Division Quality Manual located in Attachment A of the Work Plan for SWMU 2 – Dye Burial Grounds.

The U.S. Navy completes internal laboratory performance and system audits for each contracted laboratory on an 18-month schedule.

9.2.1.3 Internal Laboratory Audit Procedures

Internal systems audits are conducted to detect any problems in sample flow, analytical procedures, or documentation and to ensure adherence to laboratory SOPs. The internal audit procedures for Laucks Laboratories and Triangle Laboratories are presented in Appendix I. NSWC Crane Lab's internal audit procedures are described in SOP CR4052-PD-009.

Internal U.S. Navy laboratory audit procedures, as performed by a Navy contractor, include a pre-screening process that requires review of the laboratory's QA plan, analysis of performance evaluation samples, generation of data deliverables for those samples, an on-site technical systems audit of the laboratory, and satisfactory resolution of all deficiencies and findings.

9.2.2 External Laboratory Audits

9.2.2.1 External Laboratory Audit Responsibilities

IDEM and U.S. EPA Region 5 may perform external audits at their discretion.

Laucks Laboratories and Triangle Laboratories are also involved in various other external audits and performance evaluation studies throughout the year, as required, to maintain certifications and approvals by other regulatory agencies or programs.

9.2.2.2 External Laboratory Audit Frequency

U.S. EPA Region 5 or IDEM may conduct an external laboratory audit prior to or during sampling and analysis activities.

9.2.2.3 Overview of the External Laboratory Audit Process

External audit procedures are at the discretion of U.S. EPA Region 5 and IDEM. External laboratory audits may include (but are not limited to) review of laboratory analytical procedures, laboratory on-site audits, and submission of PE samples to the laboratory for analysis.

10.0 PREVENTIVE MAINTENANCE PROCEDURES

Equipment used to collect samples will be maintained in accordance with the manufacturer's operation and maintenance manuals. Equipment and instruments will be calibrated in accordance with the procedures and at the frequencies presented in Section 5.0 (Calibration Procedures and Frequency). Preventive maintenance for field and laboratory equipment is addressed in the remainder of this section.

10.1 FIELD EQUIPMENT PREVENTIVE MAINTENANCE

The TtNUS equipment manager and the equipment operator will be responsible for ensuring that equipment is operating properly prior to use and that routine maintenance is performed and documented. Field measurements of oxidation and reduction potential pH, specific conductance, temperature, and turbidity in ground water will be measured using an electronic instrument. Maintenance procedures for the instrument are detailed in SOP CTO 166-14. Any problems encountered while operating the instrument will be recorded in the field logbook, including a description of the symptoms and corrective actions taken. If problems with the equipment are detected and service is required, the equipment will be logged, tagged, and segregated from equipment in proper working order. Use of the equipment will not resume until the problem is corrected.

10.2 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

Proper maintenance of laboratory instruments and equipment is essential. Depending on manufacturers' recommendations, maintenance intervals are established for each instrument. All instruments will be labeled with a model number and serial number, and a maintenance logbook will be maintained for each instrument. Personnel will be alert to the maintenance status of the equipment they are using at all times. Table 10-1 provides a summary of preventive maintenance procedures performed by Laucks Testing Laboratories, Triangle Laboratories, and the NSWC Crane Laboratory for key analytical instruments and equipment associated with this project.

The use of manufacturer-recommended grades or better of supporting supplies and reagents is also a form of preventive maintenance. For example, gases used in the inductively coupled plasma (ICP) instruments will be of sufficient quality to minimize fouling of the instrument. The routine use of other supporting supplies from reputable manufacturers will help prevent unnecessary periods of instrument downtime. An inventory of critical spare parts will also be maintained by the laboratory to minimize instrument downtime.

10.3 INSPECTION/ACCEPTANCE REQUIREMENTS FOR SUPPLIES AND CONSUMABLES

All field equipment will be inspected prior to use to ensure that necessary parts are available. Most equipment planned for use in this project is simple, with few to no moving parts. Therefore, a visual inspection prior to use will be sufficient to ensure that the equipment is suitable for use. This visual inspection will occur during mobilization and during each use by the person using the equipment.

Laboratory inspection and acceptance requirements are provided in the laboratory Quality Assurance Plans. The plans present the following specifications for inspection and acceptance of supplies and consumables:

- Requirements to follow individual SOP specifications for grades of chemicals necessary to achieve acceptable analytical performance. SOPs are required to detail the necessary grade of chemicals, including compressed gases.

- Requirements to obtain primary chemical standards from reliable sources that use calibrated glassware in the preparation of the standards and to maintain all certificates supplied with the standards. Emphasis is on obtaining NIST-traceable standards where possible.

- Storage of chemical standards in accordance with applicable SOPs and in a manner that preserves their integrity.

- Routine monitoring of deionized water and other solvents to ensure that analytical systems, samples, and standards are not contaminated.

- Requirements to record the date received and the date opened on each container of chemical used for analysis.

TABLE 10-1

PREVENTIVE MAINTENANCE FOR ANALYTICAL AND SUPPORT INSTRUMENTS NSWC CRANE, CRANE, INDIANA PAGE 1 OF 2

Instrument	Preventive Maintenance	Maintenance Frequency
HPLC (Laucks, Crane)	Change Filter Frits	Monthly
	Change Column Prefilters	As needed
	Change Pump Seals	As needed
GC/MS (Laucks, Triangle)	Change Pump oil.	Every 6 months
	Change Septum	As needed
	Clean source and quadrupole rods, check power supplies in QEM box, clean inside and outside of printer, general cleaning of instrument.	As needed
	Service in accordance with manufacturer specifications under the contractor maintenance agreement.	As needed
GC (Laucks)	Change carrier and make-up gas filters	As needed
	Change trap, clean flame ionization detector (FID) jet, trim column.	As needed
ICP/MS (Laucks)	Service Intercooler.	Annually.
	Rinse and clean nebulizer cap and spray chamber.	Monthly or as needed.
	Clean torch, vacuum filters.	Bi-monthly.
	Profile instrument, examine autosampler tubing and replace as needed.	Daily.
	Empty rinse container, fill rinse water reservoir.	As needed.
Mercury Analyzer (Laucks)	Check and replace pump tubing, check and replace membrane, check and clean windows.	As needed.
Spectro-photometer (Laucks)	Clean sample compartment.	Semi-annually.
	Check wavelength calibration.	Annually.

TABLE 10-1

PREVENTIVE MAINTENANCE FOR ANALYTICAL AND SUPPORT INSTRUMENTS NSWC CRANE, CRANE, INDIANA PAGE 2 OF 2

Instrument	Preventive Maintenance	Maintenance Frequency
Ion Chromato-graph (Laucks)	Replace pump seals.	Annually.
	Lubricate analytical pump motor.	Semi-annually
	Check chromatography module and all gas lines for leaks.	Every run
	Clean conductivity detector cell electrodes, check cell calibration.	Monthly
	Replace bed supports, clean columns, clean membrane suppressor, replace autosampler pipette tip.	As needed.
TOC Analyzer (Laucks)	Replace pump tubing.	Each run.
	Change other tubing, change furnace tubes, change LiOH tube, change tin trap, adjust optical balance, change septum, change permeation dryer tubing	As needed
	Change IR filter screen, change gas tubing	Check monthly.
Refrigerators (Laucks, Triangle, Crane)	Monitor temperature	Daily.
Ovens (Laucks, Triangle, Crane)	Monitor temperature	Daily.
Balances (Laucks, Triangle, Crane)	Certify Class S weights Routine service by outside party	Annually. Annually.
	Certify and/or service in accordance with the Test, Measurement and Evaluation Division of the Army Calibration System.	Annually.
Thermometers (Laucks, Triangle, Crane)	Calibrate against NIST thermometer Recertify NIST Thermometer	Annually. Annually.
	Certify and/or service in accordance with the Test, Measurement and Evaluation Division of the Army Calibration System.	Annually.
Micropipets (Laucks, Triangle, Crane)	Check gravimetrically Clean o-rings	Monthly. As needed.

11.0 CORRECTIVE ACTION

The TtNUS QA/QC program requires that any and all personnel noting conditions adverse to quality should report these conditions immediately to the TOM and QAM. These parties, in turn, are charged with implementing appropriate corrective action in a timely manner. It is ultimately the responsibility of the QAM to document all findings and corrective actions taken and to monitor the effectiveness of the corrective measures that are performed. A brief summary of corrective actions for some specific field and laboratory QC check samples is presented in Section 3.0, Table 3-1.

11.1 FIELD CORRECTIVE ACTION

Field nonconformances or conditions adverse to quality must be identified and corrected as quickly as possible so that work integrity or product quality is not compromised. The need for corrective action may arise based on deviations from project plans and procedures, adverse field conditions, or other unforeseen circumstances. Corrective action needs may become apparent during the performance of daily work tasks or as a consequence of internal or external field audits.

Corrective action may include resampling and may involve amending previously approved field procedures. Minor modifications to field activities, such as the collection of additional samples, will be initiated at the discretion of the FOL, subject to on-site approval by NSWC Crane personnel. Major modifications, such as the elimination of a sampling point or other situations that affect compliance with or achievement of DQOs, must be approved and documented via a Field Task Modification Request (FTMR). Approval of the corrective action will be obtained by the U.S. Navy (in conjunction with U.S. EPA Region 5 and IDEM). The FOL is responsible for initiating FTMRs. A FTMR will be prepared for all deviations from the project plan documents, as applicable. An example FTMR is provided in SOP CTO 166-3 (Appendix H). Copies of all FTMRs will be maintained with the on-site project planning documents and will be placed in the final evidence file.

11.2 LABORATORY CORRECTIVE ACTION

In general, laboratory corrective actions are warranted whenever an out-of-control event or potential out-of-control event is noted. The specific corrective action taken depends on the specific analysis and the nature of the event. Generally, the following occurrences alert laboratory personnel that corrective action may be necessary:

- QC data are outside established warning or control limits.
- Method blank analyses yield concentrations of target analytes above acceptable levels.
- Undesirable trends are detected in spike %Rs or in duplicate RPDs.
- There is an unexplained change in compound detection capability.
- Inquiries concerning data quality are received.
- Deficiencies are detected by laboratory QA staff during audits or from PE sample test results.

Specific procedures for laboratory corrective actions are specified in the associated laboratory SOP. Any corrective action taken above the analyst level that cannot be performed immediately at the instrument will be documented. Corrective actions typically are documented for out-of-control situations in accordance with laboratory procedures.

Laboratory corrective actions must be documented and included as part of the final evidence file. The TtNUS TOM will be informed of all major corrective actions that do not bring DQO-related nonconformances into conformance with project DQOs. The TtNUS TOM will advise all levels of project management in accordance with Section 12.0 of this QAPP.

11.3 CORRECTIVE ACTION DURING DATA VALIDATION AND DATA ASSESSMENT

The need for corrective action may become apparent during data validation, interpretation, or presentation activities. Rework (i.e., resampling or reanalysis), a change in work procedures, or additional or refresher training are possible corrective actions relevant to data evaluation activities. The TOM will be responsible for approving the implementation of a corrective action and ensuring that it is documented appropriately. Analytical data may be qualified during data validation to alert data users of the potential that particular analytical results are potentially deficient relative to expected performance standards. Such validation practices are described in Section 8.0. When conducting data assessment for project decision making, a number of situation-dependent qualifications of data or decisions are possible. The number of possible situations or conditions precludes enumeration of all possible corrective actions; however, the approach used to identify and impose such qualifications is described in Section 8.0.

12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports to management will be provided in four primary formats during the course of this investigation: data validation reports, reports summarizing accomplishments and QA/QC issues during the field investigation, project-wide progress reports, and laboratory QA reports. The report frequencies, content, preparers, and recipients are summarized in Table 12-1.

Data validation reports will address all major and minor laboratory noncompliances, as well as noted sample matrix effects. In the event that major problems occur with the analytical laboratory (e.g., repeated or extreme holding time exceedances or calibration noncompliances, etc.), the data validation manager will notify the TOM, QAM, program manager, technical coordinator, and laboratory services coordinator. Such notifications (if necessary) are typically provided via internal memoranda and are placed in the project file. These reports contain a summary of the noncompliance, a synopsis of the impact on individual projects, and recommendations regarding corrective action and compensation adjustments. Corrective actions for major noncompliances are initiated at the program level.

The FOL will provide the TOM with daily verbal field progress reports during the course of the sampling event. These reports will explain accomplishments, deviations from the QAPP, upcoming activities, and a QA summary. The TOM provides a monthly progress report to the Navy that addresses the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.

Laucks Testing Laboratories, Triangle Laboratories, and the NSWC Crane Laboratory will provide QA reports to TtNUS if QC limits are updated or if other significant plan deviations result from unanticipated circumstances. Because MDLs will be included in the analytical data packages for the NSWC Crane Laboratory samples, it is not necessary for the laboratories to include updated MDLs in their QA reports unless the updates result in MDLs that exceed RBTLs.

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TABLE 12-1

SUMMARY OF REPORTS
NSWC CRANE, CRANE, INDIANA

Report	Content	Preparer	Frequency of Submittal	Recipient(s)
Data Validation Report	All major and minor laboratory noncompliances as well as noted sample matrix effects.	Data validation manager or designee.	Per SDG.	TOM, project file.
Major Analysis Problem Identification Report (internal memorandum)	Notification of persistent or major problems with analytical laboratory performance. Summary of the noncompliances, a synopsis of the impact on the project, and recommendations regarding corrective action and compensation adjustments.	Data validation manager or designee.	When persistent analysis problems are detected.	TOM, QAM, program manager, technical coordinator, laboratory services coordinator, project file.
Project Monthly Progress Report	Summary of the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.	TOM.	Monthly for duration of project.	Navy, project file.
Field Progress Reports	Accomplishments, deviations from the SAP, upcoming activities, and a QA summary.	FOL.	Daily, verbal, during the course of sampling.	TOM.
Laboratory QA Report	Summary of updated QC limits or significant deviations from planned activities/performance.	Laucks and Triangle Laboratories.	When QC limits are updated or when other significant plan deviations result from unanticipated circumstances.	TtNUS, project file; U.S. EPA Region 5, if changes in RLs/MDLs/IDLs cause them to exceed RBTLs or if QAPP deviations impact DQOs.

SDG = Sample delivery group.
TOM = Task order manager.
QAM = Quality assurance manager.
RL = Reporting limit.
FOL = Field operations leader.
SAP = Sampling and analysis plan.
MDL = Method detection limit.
IDL = Instrument detection limit.
DQO = Data quality objective.
QAPP = Quality assurance project plan.

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APPENDIX A

HISTORICAL DATA

SWMU 12 AND 13

BIO-REMEDIATED DATA

Concentrations of HMX, RDX, and TNT in Bioremediated Soil-Compost Deposited in Mine Fill A NSWC, Crane, Indiana

	HMX	RDX	TNT	
Residential Cleanup Goal	3,300	4	15	
Industrial Cleanup Goal	34,000	17	64	
Windrow #	mg/kg	mg/kg	mg/kg	Location Building #
31	2.2	3.7	0.3	152
43	2.9	8.3	0.3	152
44	4.7	7.2	0.3	152
45	3.4	4.4	0.3	152
46	2.8	2.9	0.3	152
51	2.1	1.2	0.2	152
64	2.6	12.4	1.8	152
75	2.0	3.5	0.2	152
76	1.8	4.3	0.3	152
77	3.7	7.9	0.4	152
78	3.9	8.0	0.5	152
59	2.0	2.8	0.4	154
61	2.1	3.6	4.5	154
66	2.1	1.9	0.3	154
68	2.2	3.0	3.6	154
70	2.1	3.0	0.4	154
72	2.1	1.7	0.2	154
73	2.0	2.4	5.9	154
74	1.9	1.5	0.4	154
79	2.5	4.0	0.2	154
80	2.2	3.3	0.5	154
81	2.1	1.8	0.2	154
82	2.6	1.8	0.2	154
83	1.7	2.4	0.5	154
84	2.5	1.8	0.2	154
85	2.3	2.3	0.4	154
86	1.9	1.4	0.2	154
87	2.3	2.1	0.2	154
88	2.6	2.3	0.2	154
89	2.2	1.7	0.7	154
90	2.4	3.5	0.2	154
91	2.4	1.1	0.3	154
94	2.8	1.9	0.2	154
95	2.1	1.3	0.2	154
96	2.8	1.3	0.2	154
97	0.9	2.8	1.9	154
99	1.1	0.5	0.3	154
100	1.6	1.9	0.2	154
192	0.3	0.7	0.5	154
194	0.5	0.6	0.6	154
195	0.5	0.4	0.5	154
197	0.3	0.2	0.5	154
198	0.5	1.1	0.4	154
3	2.2	1.1	0.3	157
5	2.2	1.1	0.3	157
12	4.9	1.6	0.4	157
33	2.0	2.8	0.7	157

Concentrations of HMX, RDX, and TNT in Bioremediated Soil-Compost Deposited in Mine Fill A NSWC, Crane, Indiana

	HMX	RDX	TNT	
Residential Cleanup Goal	3,300	4	15	
Industrial Cleanup Goal	34,000	17	64	
Windrow #	mg/kg	mg/kg	mg/kg	Location Building #
47	2.1	1.5	0.3	157
48	2.4	2.1	0.3	157
53	3.0	6.7	5.8	157
60	2.0	4.9	0.4	157
62	2.2	6.3	0.7	157
63	2.2	3.5	0.4	157
65	2.0	5.3	1.3	157
67	2.1	4.2	0.8	157
69	2.2	6.8	2.2	157
71	2.2	1.0	22.7	157
1	2.1	1.0	0.4	159
2	2.3	1.4	0.3	159
4	2.1	1.0	0.4	159
6	2.2	1.0	0.3	159
7	2.2	1.0	0.3	159
10	2.2	1.0	0.3	159
23	4.5	3.3	0.3	159
28	2.8	5.9	1.5	159
30	2.1	4.9	0.3	159
34	3.1	3.2	1.5	159
35	3.7	6.2	0.5	159
42	7.1	13.9	0.4	159
49	2.0	1.4	0.3	152 & 157
50	2.6	2.8	0.3	152 & 157
8	2.2	1.1	0.3	153/154
9	2.2	1.8	0.5	153/154
15	1.9	1.1	0.3	153/154
16	2.0	1.2	0.2	153/154
17	1.9	1.1	0.3	153/154
18	2.3	1.1	0.3	153/154
19	2.4	1.7	0.2	153/154
20	3.3	1.6	0.3	153/154
21	2.6	2.5	0.5	153/154
22	2.1	2.3	0.3	153/154
25	2.5	3.0	0.2	153/154
26	2.4	2.3	1.0	153/154
27	1.9	1.7	0.4	153/154
29	2.7	2.0	1.5	153/154
32	1.7	3.0	0.2	153/154
36	2.8	4.2	0.3	153/154
37	2.8	6.8	0.5	153/154
38	3.8	7.2	11.0	153/154
39	5.2	13.1	1.2	153/154
40	5.8	5.2	1.1	153/154
41	5.5	8.9	0.3	153/154

Concentrations of HMX, RDX, and TNT in Bioremediated Soil-Compost Deposited in Mine Fill B NSWC, Crane, Indiana

	HMX	RDX	TNT	
Residential Cleanup Goal	3,300	4	15	
Industrial Cleanup Goal	34,000	17	64	
Windrow #	mg/kg	mg/kg	mg/kg	Location Building #
143	2.3	3.8	0.6	166
144	1.6	6.8	5.5	166
145	0.5	2.7	0.6	166
146	2.1	8.2	0.7	166
147	1.8	5.5	0.5	166
150	1.0	1.2	0.9	166
169	0.7	6.6	1.1	166
179	1.0	3.8	1.9	166
199	0.7	1.9	0.4	166
120	2.0	1.9	0.4	168
122	2.6	4.6	5.2	168
123	3.3	11.1	2.9	168
124	2.0	2.2	0.3	168
126	2.5	6.7	6.7	168
138	2.0	3.4	0.4	168
141	1.5	2.9	1.2	168
180	1.4	1.9	0.5	168
187	1.1	1.6	0.5	168
177	1.6	4.2	0.5	171
182	1.2	2.0	0.6	171
189	1.4	1.5	0.5	171
183	3.1	8.7	0.5	172
184	1.9	3.3	0.5	172
106	1.6	5.7	1.0	173
108	1.9	0.9	0.2	173
117	1.8	4.1	0.8	173
105	3.7	4.9	1.9	2501
111	2.1	1.0	0.2	2501
114	2.9	3.1	0.6	2501
115	3.2	5.1	1.2	2501
116	2.5	1.6	0.2	2501
119	2.2	1.6	0.3	2501
125	2.6	6.1	0.9	2501
128	2.0	3.7	2.7	2501
190	0.7	1.9	1.6	2501
178	0.9	2.7	0.5	168,171
137	2.0	3.1	0.8	168, 171, 2501
188	1.0	2.1	0.5	168, 171, 2501
103	3.1	3.9	1.0	2501, 172
104	2.2	4.9	1.0	2501, 172
107	1.6	6.4	0.9	2501, 172
109	2.2	0.9	0.3	2501, 172
110	2.2	1.1	0.2	2501, 172
112	2.6	2.7	0.3	2501, 172
113	2.7	2.6	1.6	2501, 172
118	2.3	2.1	0.3	2501, 172
121	3.2	2.2	0.9	2501, 172

SWMU 12

POST-EXCAVATION DATA

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MPSPES05	08/29/97	1	6 - 12	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MPSPES06	08/29/97	1	12 - 18	MK	6 U	12 U	120 B	4 J	6 U	6 U	N/A
MPSPES07	08/29/97	2	6 - 12	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MPSPES08	08/29/97	2	12 - 18	MK	6 U	12 U	200 B	5 J	6 U	6 U	N/A
MFAPES009	07/22/98	148	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES010	07/22/98	148	6 - 6	MK	4 J	11 U	200 B	5 U	5 U	5 U	N/A
MFAPES011	07/22/98	147	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES012	07/22/98	147	6 - 6	MK	4 U	10 U	13 B	4 U	4 U	4 U	N/A
MFAPES013	07/22/98	146	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES013FD	07/22/98	146	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES014	07/22/98	146	6 - 6	MK	6 U	12 U	16 B	6 U	6 U	6 U	N/A
MFAPES015	07/22/98	145	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES016	07/22/98	145	6 - 6	MK	4 U	10 U	27 B	4 U	4 U	4 U	N/A
MFAPES017	07/22/98	41	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES018	07/22/98	41	6 - 6	MK	4 U	10 U	51 B	4 U	4 U	4 U	N/A
MFAPES019	07/22/98	42	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES020	07/22/98	42	6 - 6	MK	7 U	15 U	70 B	7 U	7 U	7 U	N/A
MFAPES021	07/22/98	144	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES022	07/22/98	144	6 - 6	MK	6 U	11 U	11 U	6 U	6 U	6 U	N/A
MFAPES025	08/03/98	136	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES026	08/03/98	136	6 - 6	MK	6 U	6 U	12	4 JB	6 U	6 J	N/A
MFAPES027	09/08/98	20	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES028	09/08/98	20	6 - 6	MK	5 U	11 U	10 J	5 U	5 U	5 U	N/A
MFAPES029	09/08/98	22	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES030	09/08/98	22	6 - 6	MK	5 U	11 U	15	5 U	5 U	5 U	N/A
MFAPES031	09/08/98	140	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES032	09/08/98	140	6 - 6	MK	6 U	12 U	16	6 U	6 U	6 U	N/A
MFAPES033	09/08/98	140	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES034	09/08/98	140	Sidewall	MK	5 U	11 U	11 U	5 U	5 U	5 U	N/A
MFAPES035	09/08/98	163	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES035FD	09/08/98	163	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MPSPES05	08/29/97	1	6 - 12	MK	4,800	260 U	N/A	**33,800 DJ**	250 U	650 U	250 U
MPSPES06	08/29/97	1	12 - 18	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MPSPES07	08/29/97	2	6 - 12	MK	5,030	260 U	N/A	**22,800**	250 U	650 U	250 U
MPSPES08	08/29/97	2	12 - 18	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES009	07/22/98	148	0 - 6	MK	1,450 J	260 U	250 U	2,770	250 U	650 U	250 U
MFAPES010	07/22/98	148	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES011	07/22/98	147	0 - 6	MK	2,580	260 U	250 U	***4,510***	250 U	650 U	250 U
MFAPES012	07/22/98	147	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES013	07/22/98	146	0 - 6	MK	2,560	260 U	250 U	***6,670***	250 U	650 U	250 U
MFAPES013FD	07/22/98	146	0 - 6	MK	2,680	260 U	250 U	***7,970***	250 U	650 U	250 U
MFAPES014	07/22/98	146	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES015	07/22/98	145	0 - 6	MK	2,420	260 U	250 U	***7,600***	250 U	650 U	250 U
MFAPES016	07/22/98	145	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES017	07/22/98	41	0 - 6	MK	2,700	260 U	250 U	***10,200***	250 U	650 U	103 J
MFAPES018	07/22/98	41	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES019	07/22/98	42	0 - 6	MK	2,570	260 U	250 U	***6,780***	250 U	650 U	250 U
MFAPES020	07/22/98	42	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES021	07/22/98	144	0 - 6	MK	2,550	260 U	250 U	***6,890***	250 U	650 U	250 U
MFAPES022	07/22/98	144	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES025	08/03/98	136	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES026	08/03/98	136	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES027	09/08/98	20	0 - 6	MK	1,200 J	260 U	250 U	***4,610***	250 U	650 U	250 U
MFAPES028	09/08/98	20	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES029	09/08/98	22	0 - 6	MK	822 J	260 U	250 U	2,030	250 U	650 U	168 J
MFAPES030	09/08/98	22	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES031	09/08/98	140	0 - 6	MK	1,550 J	260 U	250 U	***6,140***	250 U	650 U	250 U
MFAPES032	09/08/98	140	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES033	09/08/98	140	Sidewall	MK	4,600	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES034	09/08/98	140	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES035	09/08/98	163	0 - 6	MK	2,050 J	260 U	250 U	***10,700***	250 U	650 U	250 U
MFAPES035FD	09/08/98	163	0 - 6	MK	1,710 J	260 U	250 U	***5,860***	250 U	650 U	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MPSPES05	08/29/97	1	6 - 12	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	N/A
MPSPES06	08/29/97	1	12 - 18	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MPSPES07	08/29/97	2	6 - 12	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	N/A
MPSPES08	08/29/97	2	12 - 18	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES009	07/22/98	148	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,410,000 *
MFAPES010	07/22/98	148	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES011	07/22/98	147	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,900,000 *
MFAPES012	07/22/98	147	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES013	07/22/98	146	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,200,000 *
MFAPES013FD	07/22/98	146	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	14,200,000 *
MFAPES014	07/22/98	146	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES015	07/22/98	145	0 - 6	MK	250 U	260 U	310	250 U	250 U	250 U	250 U	11,700,000 *
MFAPES016	07/22/98	145	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES017	07/22/98	41	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,200,000 *
MFAPES018	07/22/98	41	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES019	07/22/98	42	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,500,000 *
MFAPES020	07/22/98	42	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES021	07/22/98	144	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10,800,000 *
MFAPES022	07/22/98	144	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES025	08/03/98	136	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,900,000
MFAPES026	08/03/98	136	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES027	09/08/98	20	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,500,000
MFAPES028	09/08/98	20	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES029	09/08/98	22	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,500,000
MFAPES030	09/08/98	22	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES031	09/08/98	140	0 - 6	MK	250 U	260 U	67.400 J	250 U	250 U	250 U	250 U	11,600,000
MFAPES032	09/08/98	140	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES033	09/08/98	140	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	19,700,000
MFAPES034	09/08/98	140	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES035	09/08/98	163	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,200,000
MFAPES035FD	09/08/98	163	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,800,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MPSPES05	08/29/97	1	6 - 12	MK	N/A	89,100	470 B	16,200	14,700	N/A	N/A	N/A
MPSPES06	08/29/97	1	12 - 18	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MPSPES07	08/29/97	2	6 - 12	MK	N/A	95,700	390 B	14,700	14,700	N/A	N/A	N/A
MPSPES08	08/29/97	2	12 - 18	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES009	07/22/98	148	0 - 6	MK	N/A	48,500	30 U	11,200	9,600 B	40 NU	N/A	N/A
MFAPES010	07/22/98	148	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES011	07/22/98	147	0 - 6	MK	N/A	68,100	40 U	16,400	11,500 B	40 NU	N/A	N/A
MFAPES012	07/22/98	147	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES013	07/22/98	146	0 - 6	MK	N/A	61,800	30 U	13,200	10,700 B	40 NU	N/A	N/A
MFAPES013FD	07/22/98	146	0 - 6	MK	N/A	56,300	30 U	16,700	10,600 B	70 N	N/A	N/A
MFAPES014	07/22/98	146	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES015	07/22/98	145	0 - 6	MK	N/A	95,000	30 U	18,000	11,200 B	40 NU	N/A	N/A
MFAPES016	07/22/98	145	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES017	07/22/98	41	0 - 6	MK	N/A	75,500	30 U	20,400	11,400	40 NU	N/A	N/A
MFAPES018	07/22/98	41	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES019	07/22/98	42	0 - 6	MK	N/A	84,600	30 U	16,500	9,600 B	40 NU	N/A	N/A
MFAPES020	07/22/98	42	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES021	07/22/98	144	0 - 6	MK	N/A	50,900	30 U	12,900	9,000 B	40 NU	N/A	N/A
MFAPES022	07/22/98	144	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES025	08/03/98	136	0 - 6	MK	N/A	79,900	290 B	20,900	9,700 B	40 U	N/A	N/A
MFAPES026	08/03/98	136	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES027	09/08/98	20	0 - 6	MK	N/A	295,000	40 U	17,500 N*	9,500 B	40 NU	N/A	N/A
MFAPES028	09/08/98	20	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES029	09/08/98	22	0 - 6	MK	N/A	80,300	30	21,100 N*	36,800	40 NU	N/A	N/A
MFAPES030	09/08/98	22	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES031	09/08/98	140	0 - 6	MK	N/A	189,000	30 U	18,900 N*	10,400 B	40 NU	N/A	N/A
MFAPES032	09/08/98	140	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES033	09/08/98	140	Sidewall	MK	N/A	65,300	30 U	24,200 N*	14,500 B	120 N	N/A	N/A
MFAPES034	09/08/98	140	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES035	09/08/98	163	0 - 6	MK	N/A	48,400	30	33,100 N*	12,800 B	70 N	N/A	N/A
MFAPES035FD	09/08/98	163	0 - 6	MK	N/A	48,700	30 U	25,300 N*	14,700 B	40 NU	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES036	09/08/98	163	6 - 6	MK	4 U	10 U	21	4 U	4 U	4 U	N/A
MFAPES037	09/09/98	163	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES038	09/09/98	163	Sidewall	MK	4 U	10 U	7 J	4 U	4 U	5 U	N/A
MFAPES039	09/09/98	164	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES040	09/09/98	164	6 - 6	MK	4 U	9 U	27	4 U	4 U	4 U	N/A
MFAPES041	09/09/98	164	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES042	09/09/98	164	Sidewall	MK	5 U	9 U	44	5 U	5 U	5 U	N/A
MFAPES043	09/09/98	165	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES044	09/09/98	165	6 - 6	MK	4 U	9 U	14	4 U	4 U	4 U	N/A
MFAPES045	09/09/98	165	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES046	09/09/98	165	Sidewall	MK	4 U	9 U	14	4 U	4 U	4 U	N/A
MFAPES047	09/10/98	176	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES048	09/10/98	176	6 - 6	MK	5 U	5 U	52	5 U	5 U	5 U	N/A
MFAPES049	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES050	09/10/98	176	Sidewall	MK	5 U	5 U	29	5 U	5 U	5 U	N/A
MFAPES051	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES052	09/10/98	176	Sidewall	MK	10 U	10 U	10 U	10 U	10 U	10 U	N/A
MFAPES053	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES054	09/10/98	176	Sidewall	MK	6 U	6 U	35	6 U	6 U	6 U	N/A
MFAPES055	09/10/98	166	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES056	09/10/98	166	6 - 6	MK	5 U	5 U	3 J	5 U	5 U	5 U	N/A
MFAPES057	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES058	09/14/98	166	Sidewall	MK	7 U	7 U	7 U	7 U	7 U	7 U	N/A
MFAPES059	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES060	09/14/98	166	Sidewall	MK	5 U	5 U	2 J	5	5 U	5 U	N/A
MFAPES061	09/14/98	167	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES062	09/14/98	167	6 - 6	MK	5 U	5 U	12	5 J	5 U	5 U	N/A
MFAPES063	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES064	09/14/98	167	Sidewall	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES065	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES066	09/14/98	167	Sidewall	MK	6 U	6 U	10	6	6 U	6 U	N/A
MFAPES067	09/30/98	18	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES036	09/08/98	163	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES037	09/09/98	163	Sidewall	MK	5,150	260 U	250 U	227 JP	250 U	650 U	250 U
MFAPES038	09/09/98	163	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES039	09/09/98	164	0 - 6	MK	3,350	260 U	250 U	***10,600***	250 U	650 U	250 U
MFAPES040	09/09/98	164	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES041	09/09/98	164	Sidewall	MK	6,760	260 U	250 U	549 J	250 U	650 U	250 U
MFAPES042	09/09/98	164	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES043	09/09/98	165	0 - 6	MK	2,740	260 U	250 U	***4,570***	250 U	650 U	250 U
MFAPES044	09/09/98	165	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES045	09/09/98	165	Sidewall	MK	3,830	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES046	09/09/98	165	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES047	09/10/98	176	0 - 6	MK	3,300	260 U	250 U	***4,560***	250 U	650 U	250 U
MFAPES048	09/10/98	176	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES049	09/10/98	176	Sidewall	MK	8,950	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES050	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES051	09/10/98	176	Sidewall	MK	2,040 J	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES052	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES053	09/10/98	176	Sidewall	MK	6,050	260 U	250 U	115 JP	250 U	650 U	250 U
MFAPES054	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES055	09/10/98	166	0 - 6	MK	3,860	260 U	250 U	***5,860***	250 U	650 U	250 U
MFAPES056	09/10/98	166	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES057	09/14/98	166	Sidewall	MK	8,340	260 U	250 U	**25,300**	250 U	650 U	250 U
MFAPES058	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES059	09/14/98	166	Sidewall	MK	3,900	260 U	250 U	***5,570***	250 U	650 U	250 U
MFAPES060	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES061	09/14/98	167	0 - 6	MK	1,770 J	260 U	250 U	***4,300***	250 U	650 U	250 U
MFAPES062	09/14/98	167	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES063	09/14/98	167	Sidewall	MK	7,320	260 U	250 U	***4,140***	250 U	650 U	250 U
MFAPES064	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES065	09/14/98	167	Sidewall	MK	5,470	260 U	250 U	***13,100***	250 U	650 U	250 U
MFAPES066	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES067	09/30/98	18	0 - 6	MK	2,290	260 U	250 U	***5,880***	250 U	650 U	306

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES036	09/08/98	163	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES037	09/09/98	163	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	18,300,000
MFAPES038	09/09/98	163	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES039	09/09/98	164	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,000,000
MFAPES040	09/09/98	164	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES041	09/09/98	164	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	16,200,000
MFAPES042	09/09/98	164	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES043	09/09/98	165	0 - 6	MK	250 U	260 U	252	89.100 J	250 U	250 U	250 U	9,860,000
MFAPES044	09/09/98	165	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES045	09/09/98	165	Sidewall	MK	250 U	260 U	57.700 J	133 J	250 U	250 U	250 U	16,600,000
MFAPES046	09/09/98	165	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES047	09/10/98	176	0 - 6	MK	250 U	260 U	52.400 J	73.800 J	250 U	250 U	250 U	11,000,000
MFAPES048	09/10/98	176	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES049	09/10/98	176	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,400,000
MFAPES050	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES051	09/10/98	176	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	19,200,000
MFAPES052	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES053	09/10/98	176	Sidewall	MK	250 U	260 U	54.800 J	101 J	250 U	250 U	250 U	15,800,000
MFAPES054	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES055	09/10/98	166	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	9,370,000
MFAPES056	09/10/98	166	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES057	09/14/98	166	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	14,400,000
MFAPES058	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES059	09/14/98	166	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,100,000
MFAPES060	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES061	09/14/98	167	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	9,440,000
MFAPES062	09/14/98	167	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES063	09/14/98	167	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	9,480,000
MFAPES064	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES065	09/14/98	167	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,770,000
MFAPES066	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES067	09/30/98	18	0 - 6	MK	250 U	260 U	127 J	210 J	250 U	250 U	250 U	8,020,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES036	09/08/98	163	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES037	09/09/98	163	Sidewall	MK	N/A	71,100	200 B	24,500	18,100 B	60 N	N/A	N/A
MFAPES038	09/09/98	163	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES039	09/09/98	164	0 - 6	MK	N/A	37,700	210 B	32,800	14,100 B	40 NU	N/A	N/A
MFAPES040	09/09/98	164	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES041	09/09/98	164	Sidewall	MK	N/A	61,000	150 B	19,900	14,100 B	120 N	N/A	N/A
MFAPES042	09/09/98	164	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES043	09/09/98	165	0 - 6	MK	N/A	43,300	140 B	14,300	12,500 B	40 NU	N/A	N/A
MFAPES044	09/09/98	165	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES045	09/09/98	165	Sidewall	MK	N/A	62,300	220 B	29,800	19,300 B	40 NU	N/A	N/A
MFAPES046	09/09/98	165	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES047	09/10/98	176	0 - 6	MK	N/A	40,600	30 U	22,100	12,400 ENB	40 U	N/A	N/A
MFAPES048	09/10/98	176	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES049	09/10/98	176	Sidewall	MK	N/A	121,000	30 U	55,100	41,300 EN	40	N/A	N/A
MFAPES050	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES051	09/10/98	176	Sidewall	MK	N/A	63,800	30 U	20,800	17,100 ENB	40 U	N/A	N/A
MFAPES052	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES053	09/10/98	176	Sidewall	MK	N/A	51,000	160 B	18,900	18,200 ENB	70	N/A	N/A
MFAPES054	09/10/98	176	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES055	09/10/98	166	0 - 6	MK	N/A	37,600	30 U	14,100	10,000 ENB	40 U	N/A	N/A
MFAPES056	09/10/98	166	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES057	09/14/98	166	Sidewall	MK	N/A	53,900	260 B	19,400	12,700 B	40 U	N/A	N/A
MFAPES058	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES059	09/14/98	166	Sidewall	MK	N/A	48,700	200 B	18,200	9,600 B	40 U	N/A	N/A
MFAPES060	09/14/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES061	09/14/98	167	0 - 6	MK	N/A	36,900	200 B	16,200	9,000 B	40 U	N/A	N/A
MFAPES062	09/14/98	167	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES063	09/14/98	167	Sidewall	MK	N/A	27,700	200 B	16,000	11,000 B	40 U	N/A	N/A
MFAPES064	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES065	09/14/98	167	Sidewall	MK	N/A	24,700	470 B	12,000	10,000 B	40 U	N/A	N/A
MFAPES066	09/14/98	167	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES067	09/30/98	18	0 - 6	MK	N/A	51,800	30 U	11,200	8,800 B	70	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES067FD	09/30/98	18	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES068	09/30/98	18	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES069	09/30/98	17	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES070	09/30/98	17	6 - 6	MK	5 U	5 U	5 U	4 J	5 U	5 U	N/A
MFAPES071	09/30/98	8	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES072	09/30/98	8	6 - 6	MK	5 U	5 U	5 U	8	5 U	5 U	N/A
MFAPES073	09/30/98	139	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES074	09/30/98	139	6 - 6	MK	5 U	5 U	11 B	8	5 U	5 U	N/A
MFAPES075	10/01/98	7	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES076	10/01/98	7	6 - 6	MK	5 U	5 U	44	3 J	5 U	5 U	N/A
MFAPES077	10/01/98	6	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES078	10/01/98	6	6 - 6	MK	6 U	6 U	22	4 J	6 U	6 U	N/A
MFAPES079	10/01/98	9	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES080	10/01/98	9	6 - 6	MK	5 U	5 U	45	4 J	5 U	5 U	N/A
MFAPES081	10/01/98	16	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES082	10/01/98	16	6 - 6	MK	5 U	5 U	46	4 J	5 U	5 U	N/A
MFAPES083	10/01/98	138	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES084	10/01/98	138	6 - 6	MK	6 U	6 U	6 U	4 J	6 U	6 U	N/A
MFAPES085	10/01/98	137	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES086	10/01/98	137	6 - 6	MK	5 U	5 U	26 B	9	5 U	5 U	N/A
MFAPES088	10/13/98	200	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES088FD	10/13/98	200	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES089	10/13/98	200	6 - 6	MK	5 U	5 U	38	2 J	5 U	5 U	N/A
MFAPES090	10/13/98	200	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES091	10/13/98	201	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES092	10/13/98	201	6 - 6	MK	5 U	5 U	35	3 J	5 U	5 U	N/A
MFAPES093	10/14/98	122	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES094	10/14/98	122	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES095	10/14/98	127	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES096	10/14/98	127	6 - 6	MK	5 U	5 U	21	5 U	5 U	5 U	N/A
MFAPES097	10/14/98	128	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES067FD	09/30/98	18	0 - 6	MK	2,330	260 U	250 U	***6,480***	250 U	650 U	113 J
MFAPES068	09/30/98	18	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES069	09/30/98	17	0 - 6	MK	8,820	260 U	250 U	**61,800 D**	250 U	650 U	306
MFAPES070	09/30/98	17	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES071	09/30/98	8	0 - 6	MK	4,360	260 U	250 U	***11,100***	250 U	650 U	201 J
MFAPES072	09/30/98	8	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES073	09/30/98	139	0 - 6	MK	952 J	260 U	250 U	***4,890***	250 U	650 U	135 J
MFAPES074	09/30/98	139	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES075	10/01/98	7	0 - 6	MK	1,460 J	260 U	250 U	3,410	250 U	650 U	364
MFAPES076	10/01/98	7	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES077	10/01/98	6	0 - 6	MK	1,070 J	260 U	250 U	***6,540***	250 U	650 U	250 U
MFAPES078	10/01/98	6	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES079	10/01/98	9	0 - 6	MK	1,710 J	260 U	250 U	3,370	250 U	650 U	271
MFAPES080	10/01/98	9	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES081	10/01/98	16	0 - 6	MK	949 J	260 U	250 U	***7,000***	250 U	650 U	65.4 J
MFAPES082	10/01/98	16	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES083	10/01/98	138	0 - 6	MK	1,740 J	260 U	250 U	***5,240***	250 U	650 U	104 J
MFAPES084	10/01/98	138	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES085	10/01/98	137	0 - 6	MK	1,940 J	260 U	250 U	***11,500***	250 U	650 U	250 U
MFAPES086	10/01/98	137	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES088	10/13/98	200	0 - 6	MK	11,400	260 U	250 U	**22,800**	250 U	650 U	267
MFAPES088FD	10/13/98	200	0 - 6	MK	9,130	260 U	250 U	19,400	250 U	650 U	314 P
MFAPES089	10/13/98	200	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES090	10/13/98	200	Sidewall	MK	1,220 J	260 U	250 U	**17,500**	250 U	650 U	250 U
MFAPES091	10/13/98	201	0 - 6	MK	1,540 J	248 U	238 U	***7,250***	238 U	619 U	1,340
MFAPES092	10/13/98	201	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES093	10/14/98	122	0 - 6	MK	3,690	260 U	250 U	***16,000***	250 U	650 U	140 J
MFAPES094	10/14/98	122	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES095	10/14/98	127	0 - 6	MK	27,500	260 U	250 U	**GT 103,000 E**	250 U	650 U	285 P
MFAPES096	10/14/98	127	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES097	10/14/98	128	0 - 6	MK	15,500	260 U	250 U	**108,000 D**	250 U	650 U	237 J

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES067FD	09/30/98	18	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	N/A
MFAPES068	09/30/98	18	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES069	09/30/98	17	0 - 6	MK	250 U	260 U	102 J	230 J	250 U	250 U	250 U	12,300,000
MFAPES070	09/30/98	17	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES071	09/30/98	8	0 - 6	MK	250 U	457 P	250 U	250 U	250 U	250 U	250 U	5,190,000
MFAPES072	09/30/98	8	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES073	09/30/98	139	0 - 6	MK	250 U	260 U	160 J	92.300 J	250 U	250 U	250 U	6,360,000
MFAPES074	09/30/98	139	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES075	10/01/98	7	0 - 6	MK	250 U	260 U	189 J	216 J	250 U	250 U	250 U	6,730,000
MFAPES076	10/01/98	7	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES077	10/01/98	6	0 - 6	MK	250 U	260 U	202 J	250 U	250 U	250 U	250 U	6,370,000
MFAPES078	10/01/98	6	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES079	10/01/98	9	0 - 6	MK	250 U	260 U	459	382	250 U	250 U	250 U	6,860,000
MFAPES080	10/01/98	9	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES081	10/01/98	16	0 - 6	MK	250 U	260 U	123 J	250 U	250 U	250 U	250 U	6,450,000
MFAPES082	10/01/98	16	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES083	10/01/98	138	0 - 6	MK	250 U	260 U	237 J	128 J	250 U	250 U	250 U	6,030,000
MFAPES084	10/01/98	138	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES085	10/01/98	137	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	6,330,000
MFAPES086	10/01/98	137	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES088	10/13/98	200	0 - 6	MK	250 U	178 J	250 U	250 U	250 U	250 U	250 U	7,820,000
MFAPES088FD	10/13/98	200	0 - 6	MK	250 U	260 U	214 JP	254	250 U	250 U	250 U	N/A
MFAPES089	10/13/98	200	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES090	10/13/98	200	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	9,540,000
MFAPES091	10/13/98	201	0 - 6	MK	238 U	248 U	189 J	237 J	238 U	238 U	238 U	3,310,000
MFAPES092	10/13/98	201	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES093	10/14/98	122	0 - 6	MK	250 U	260 U	107 J	81.800 J	250 U	250 U	250 U	13,900,000
MFAPES094	10/14/98	122	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES095	10/14/98	127	0 - 6	MK	250 U	260 U	169 J	236 JP	250 U	250 U	250 U	10,500,000
MFAPES096	10/14/98	127	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES097	10/14/98	128	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,300,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES067FD	09/30/98	18	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES068	09/30/98	18	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES069	09/30/98	17	0 - 6	MK	N/A	54,000	30 U	15,700	11,800 B	160	N/A	N/A
MFAPES070	09/30/98	17	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES071	09/30/98	8	0 - 6	MK	N/A	40,300	30 U	7,300	7,400 B	50	N/A	N/A
MFAPES072	09/30/98	8	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES073	09/30/98	139	0 - 6	MK	N/A	61,100	30 U	28,000	10,300 B	50	N/A	N/A
MFAPES074	09/30/98	139	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES075	10/01/98	7	0 - 6	MK	N/A	32,000	50 B	9,700	7,300 B	200 N*	N/A	N/A
MFAPES076	10/01/98	7	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES077	10/01/98	6	0 - 6	MK	N/A	44,100	40 B	10,400	7,600 B	80 N*	N/A	N/A
MFAPES078	10/01/98	6	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES079	10/01/98	9	0 - 6	MK	N/A	41,700	70 B	14,300	12,000 B	40 N*U	N/A	N/A
MFAPES080	10/01/98	9	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES081	10/01/98	16	0 - 6	MK	N/A	78,400	30 U	19,000	9,100 B	40 N*U	N/A	N/A
MFAPES082	10/01/98	16	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES083	10/01/98	138	0 - 6	MK	N/A	53,200	30 U	12,800	13,000 B	110 N*	N/A	N/A
MFAPES084	10/01/98	138	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES085	10/01/98	137	0 - 6	MK	N/A	163,000	30 U	13,300	9,500 B	160 N*	N/A	N/A
MFAPES086	10/01/98	137	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES088	10/13/98	200	0 - 6	MK	N/A	90,500	240 B	9,100 N	10,600 B	20 U	N/A	N/A
MFAPES088FD	10/13/98	200	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES089	10/13/98	200	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES090	10/13/98	200	Sidewall	MK	N/A	60,500	40 U	11,000 N	12,700 B	80	N/A	N/A
MFAPES091	10/13/98	201	0 - 6	MK	N/A	36,200	30 U	4,400 N	3,700 B	20 U	N/A	N/A
MFAPES092	10/13/98	201	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES093	10/14/98	122	0 - 6	MK	N/A	53,700	40 U	19,000	13,100 B	40	N/A	N/A
MFAPES094	10/14/98	122	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES095	10/14/98	127	0 - 6	MK	N/A	223,000	30 U	15,400	12,000 B	40	N/A	N/A
MFAPES096	10/14/98	127	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES097	10/14/98	128	0 - 6	MK	N/A	93,000	40 U	17,100	14,800 B	40	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES098	10/14/98	128	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES099	10/14/98	13	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES100	10/14/98	13	6 - 6	MK	5 U	5 U	11	10	5 U	5 U	N/A
MFAPES101	10/14/98	14	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES102	10/14/98	14	6 - 6	MK	5 U	5 U	21	5 U	5 U	5 U	N/A
MFAPES103	10/15/98	123	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES104	10/15/98	123	6 - 6	MK	5 U	5 U	21	5 U	5 U	5 U	N/A
MFAPES105	10/15/98	124	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES106	10/15/98	124	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES107	10/15/98	125	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES107FD	10/15/98	125	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES108	10/15/98	125	6 - 6	MK	5 U	5 U	68	5 U	5 U	5 U	N/A
MFAPES109	10/15/98	126	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES110	10/15/98	126	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES111	10/15/98	3	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES112	10/15/98	3	6 - 6	MK	5 U	5 U	70	5 U	5 U	5 U	N/A
MFAPES113	10/15/98	4	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES114	10/15/98	4	6 - 6	MK	5 U	5 U	16	5 U	5 U	5	N/A
MFAPES115	10/15/98	4	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES116	10/15/98	4	Sidewall	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES117	10/15/98	134	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES118	10/15/98	134	6 - 6	MK	6 U	6 U	17	6 U	6 U	6 U	N/A
MFAPES119	10/20/98	134	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES120	10/20/98	134	Sidewall	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES121	10/20/98	5	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES122	10/20/98	5	6 - 6	MK	5 U	5 U	6	5 U	5 U	5 U	N/A
MFAPES123	10/20/98	202	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES124	10/20/98	202	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES125	10/20/98	203	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES126	10/20/98	203	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES098	10/14/98	128	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES099	10/14/98	13	0 - 6	MK	457 JP	260 U	250 U	1,760	250 U	650 U	250 U
MFAPES100	10/14/98	13	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES101	10/14/98	14	0 - 6	MK	1,220 J	260 U	250 U	***6,100***	250 U	650 U	250 U
MFAPES102	10/14/98	14	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES103	10/15/98	123	0 - 6	MK	1,980 J	248 U	238 U	***5,270***	238 U	619 U	238 U
MFAPES104	10/15/98	123	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES105	10/15/98	124	0 - 6	MK	6,260	260 U	250 U	***8,210***	250 U	650 U	103 J
MFAPES106	10/15/98	124	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES107	10/15/98	125	0 - 6	MK	1,210 J	260 U	250 U	3,700	250 U	650 U	250 U
MFAPES107FD	10/15/98	125	0 - 6	MK	837 J	248 U	238 U	2,690	238 U	619 U	238 U
MFAPES108	10/15/98	125	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES109	10/15/98	126	0 - 6	MK	8,940	260 U	250 U	**28,900**	250 U	650 U	250 U
MFAPES110	10/15/98	126	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES111	10/15/98	3	0 - 6	MK	45,500	248 U	238 U	**428,000 D**	238 U	619 U	8,110
MFAPES112	10/15/98	3	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES113	10/15/98	4	0 - 6	MK	107,000 DJ	260 U	250 U	**1,000,000 D**	250 U	650 U	7,140
MFAPES114	10/15/98	4	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES115	10/15/98	4	Sidewall	MK	67,500	260 U	250 U	**555,000 D**	250 U	650 U	2,660
MFAPES116	10/15/98	4	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES117	10/15/98	134	0 - 6	MK	171,000 DJ	248 U	238 U	**1,530,000 D**	238 U	619 U	618
MFAPES118	10/15/98	134	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES119	10/20/98	134	Sidewall	MK	GT 326,000 E	248 U	238 U	**GT 1,940,000 E**	238 U	619 U	2,770
MFAPES120	10/20/98	134	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES121	10/20/98	5	0 - 6	MK	20,000	260 U	250 U	**GT 246,000 E**	250 U	650 U	14,200
MFAPES122	10/20/98	5	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES123	10/20/98	202	0 - 6	MK	10,400	260 U	250 U	**GT 55,200 E**	250 U	650 U	656
MFAPES124	10/20/98	202	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES125	10/20/98	203	0 - 6	MK	3,550	260 U	250 U	**24,800**	250 U	650 U	1,730
MFAPES126	10/20/98	203	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES098	10/14/98	128	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES099	10/14/98	13	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	16,100,000
MFAPES100	10/14/98	13	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES101	10/14/98	14	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,400,000
MFAPES102	10/14/98	14	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES103	10/15/98	123	0 - 6	MK	238 U	248 U	238 U	238 U	238 U	238 U	238 U	11,400,000
MFAPES104	10/15/98	123	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES105	10/15/98	124	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,200,000
MFAPES106	10/15/98	124	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES107	10/15/98	125	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10,400,000
MFAPES107FD	10/15/98	125	0 - 6	MK	238 U	248 U	238 U	238 U	238 U	238 U	238 U	11,900,000
MFAPES108	10/15/98	125	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES109	10/15/98	126	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10,500,000
MFAPES110	10/15/98	126	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES111	10/15/98	3	0 - 6	MK	238 U	248 U	1,990	2,860	238 U	238 U	238 U	12,700,000
MFAPES112	10/15/98	3	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES113	10/15/98	4	0 - 6	MK	250 U	260 U	2,330	2,990	250 U	250 U	250 U	12,100,000
MFAPES114	10/15/98	4	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES115	10/15/98	4	Sidewall	MK	250 U	260 U	822	5,800	250 U	250 U	250 U	10,800,000
MFAPES116	10/15/98	4	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES117	10/15/98	134	0 - 6	MK	238 U	248 U	381	2,000	238 U	238 U	238 U	10,600,000
MFAPES118	10/15/98	134	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES119	10/20/98	134	Sidewall	MK	238 U	248 U	379	1,090	238 U	238 U	238 U	12,300,000
MFAPES120	10/20/98	134	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES121	10/20/98	5	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,000,000
MFAPES122	10/20/98	5	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES123	10/20/98	202	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	9,100,000
MFAPES124	10/20/98	202	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES125	10/20/98	203	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,400,000
MFAPES126	10/20/98	203	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES098	10/14/98	128	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES099	10/14/98	13	0 - 6	MK	N/A	58,300	40 U	20,100	14,900 B	60	N/A	N/A
MFAPES100	10/14/98	13	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES101	10/14/98	14	0 - 6	MK	N/A	43,300	40 U	18,600	13,700 B	40 U	N/A	N/A
MFAPES102	10/14/98	14	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES103	10/15/98	123	0 - 6	MK	N/A	47,900	40 U	17,700	11,300 B	40 U	N/A	N/A
MFAPES104	10/15/98	123	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES105	10/15/98	124	0 - 6	MK	N/A	58,500	70 B	15,900	10,100 B	40 U	N/A	N/A
MFAPES106	10/15/98	124	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES107	10/15/98	125	0 - 6	MK	N/A	48,700	30 U	15,300	10,000 B	40 U	N/A	N/A
MFAPES107FD	10/15/98	125	0 - 6	MK	N/A	56,700	40 U	18,700	13,100 B	40 U	N/A	N/A
MFAPES108	10/15/98	125	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES109	10/15/98	126	0 - 6	MK	N/A	106,000	30 U	16,500	12,500 B	40 U	N/A	N/A
MFAPES110	10/15/98	126	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES111	10/15/98	3	0 - 6	MK	N/A	68,100	50 B	17,900	11,600 B	40 U	N/A	N/A
MFAPES112	10/15/98	3	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES113	10/15/98	4	0 - 6	MK	N/A	69,300	70 B	23,800	15,100 B	40 U	N/A	N/A
MFAPES114	10/15/98	4	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES115	10/15/98	4	Sidewall	MK	N/A	69,800	100 B	14,300	17,700 B	40 U	N/A	N/A
MFAPES116	10/15/98	4	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES117	10/15/98	134	0 - 6	MK	N/A	45,000	90 B	21,200	13,700 B	40 U	N/A	N/A
MFAPES118	10/15/98	134	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES119	10/20/98	134	Sidewall	MK	N/A	58,800	120 B	16,100 N	14,900 B	20 *B	N/A	N/A
MFAPES120	10/20/98	134	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES121	10/20/98	5	0 - 6	MK	N/A	48,700	100 B	20,800 N	9,300 B	30 *B	N/A	N/A
MFAPES122	10/20/98	5	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES123	10/20/98	202	0 - 6	MK	N/A	46,900	180 B	22,500 N	10,000 B	30 *B	N/A	N/A
MFAPES124	10/20/98	202	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES125	10/20/98	203	0 - 6	MK	N/A	102,000	160 B	17,500 N	20,700 B	50 *	N/A	N/A
MFAPES126	10/20/98	203	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES127	10/20/98	203	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES127FD	10/20/98	203	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES128	10/20/98	203	Sidewall	MK	5 U	5 U	30	5 U	3 J	5 U	N/A
MFAPES129	10/20/98	45	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES130	10/20/98	45	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES131	10/20/98	48	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES132	10/20/98	48	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES133	10/20/98	50	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES134	10/20/98	50	6 - 6	MK	5 U	5 U	78	5 U	5 U	5 U	N/A
MFAPES135	10/20/98	52	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES136	10/20/98	52	6 - 6	MK	5 U	5 U	16	5 U	5 U	5 U	N/A
MFAPES137	10/22/98	54	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES138	10/22/98	54	6 - 6	MK	5 U	5 U	5 U	7 B	5 U	5 U	N/A
MFAPES139	10/22/98	56	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES140	10/22/98	56	6 - 6	MK	6 U	6 U	6 U	10 B	6 U	6 U	N/A
MFAPES141	10/22/98	49	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES142	10/22/98	49	6 - 6	MK	5 U	5 U	5 U	6 B	5 U	5 U	N/A
MFAPES143	10/22/98	43	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES144	10/22/98	43	6 - 6	MK	5 U	5 U	5 U	8 B	5 U	5 U	N/A
MFAPES145	10/23/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES146	10/26/98	166	Sidewall	MK	6 U	6 U	190	6 U	6 U	6 U	N/A
MFAPES147	10/26/98	143	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES147FD	10/26/98	143	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES148	10/26/98	143	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES149	10/28/98	15	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES150	10/28/98	15	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES151	10/28/98	142	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES152	10/28/98	142	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES153	10/28/98	1	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES154	10/28/98	1	6 - 6	MK	6 U	6 U	6 U	21	6 U	6 U	N/A
MFAPES155	10/26/98	2	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES127	10/20/98	203	Sidewall	MK	5,910	248 U	238 U	**25,200**	238 U	619 U	3,740
MFAPES127FD	10/20/98	203	Sidewall	MK	8,880	260 U	250 U	**GT 55,400 E**	250 U	650 U	6,640
MFAPES128	10/20/98	203	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES129	10/20/98	45	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES130	10/20/98	45	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES131	10/20/98	48	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES132	10/20/98	48	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES133	10/20/98	50	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES134	10/20/98	50	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES135	10/20/98	52	0 - 6	MK	968 J	260 U	250 U	3,230	250 U	650 U	350
MFAPES136	10/20/98	52	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES137	10/22/98	54	0 - 6	MK	4,260	260 U	250 U	***11,600***	250 U	650 U	648
MFAPES138	10/22/98	54	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES139	10/22/98	56	0 - 6	MK	23,400	260 U	250 U	**162,000 D**	250 U	650 U	***46,600 D***
MFAPES140	10/22/98	56	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES141	10/22/98	49	0 - 6	MK	4,160	260 U	250 U	***11,500***	250 U	650 U	389
MFAPES142	10/22/98	49	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES143	10/22/98	43	0 - 6	MK	6,690	260 U	250 U	***6,220***	250 U	650 U	250 U
MFAPES144	10/22/98	43	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES145	10/23/98	166	Sidewall	MK	12,000	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES146	10/26/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES147	10/26/98	143	0 - 6	MK	6,140	260 U	250 U	***5,530***	250 U	650 U	250 U
MFAPES147FD	10/26/98	143	0 - 6	MK	5,030	260 U	250 U	4,760	250 U	650 U	250 U
MFAPES148	10/26/98	143	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES149	10/28/98	15	0 - 6	MK	5,540	260 U	250 U	***9,510***	250 U	650 U	245 JP
MFAPES150	10/28/98	15	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES151	10/28/98	142	0 - 6	MK	4,120	260 U	250 U	***15,400***	250 U	650 U	152 JP
MFAPES152	10/28/98	142	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES153	10/28/98	1	0 - 6	MK	5,600	260 U	250 U	***9,360***	250 U	650 U	203 J
MFAPES154	10/28/98	1	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES155	10/26/98	2	0 - 6	MK	9,120	260 U	250 U	**51,400 D**	250 U	650 U	2,980

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES127	10/20/98	203	Sidewall	MK	238 U	248 U	742	238 U	238 U	238 U	238 U	10,100,000
MFAPES127FD	10/20/98	203	Sidewall	MK	250 U	260 U	1,030	250 U	250 U	250 U	250 U	14,500,000
MFAPES128	10/20/98	203	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES129	10/20/98	45	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,200,000
MFAPES130	10/20/98	45	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES131	10/20/98	48	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,700,000
MFAPES132	10/20/98	48	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES133	10/20/98	50	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,300,000
MFAPES134	10/20/98	50	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES135	10/20/98	52	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,700,000
MFAPES136	10/20/98	52	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES137	10/22/98	54	0 - 6	MK	250 U	260 U	788	608	250 U	250 U	250 U	15,100,000
MFAPES138	10/22/98	54	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES139	10/22/98	56	0 - 6	MK	250 U	260 U	1,200	1,360	250 U	250 U	250 U	12,100,000
MFAPES140	10/22/98	56	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES141	10/22/98	49	0 - 6	MK	250 U	260 U	115 J	250 U	250 U	250 U	250 U	7,030,000
MFAPES142	10/22/98	49	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES143	10/22/98	43	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10,500,000
MFAPES144	10/22/98	43	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES145	10/23/98	166	Sidewall	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15500000 *
MFAPES146	10/26/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES147	10/26/98	143	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12300000 *
MFAPES147FD	10/26/98	143	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10700000 *
MFAPES148	10/26/98	143	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES149	10/28/98	15	0 - 6	MK	250 U	260 U	331	304	250 U	250 U	250 U	16,000,000
MFAPES150	10/28/98	15	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES151	10/28/98	142	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,200,000
MFAPES152	10/28/98	142	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES153	10/28/98	1	0 - 6	MK	250 U	260 U	290	219 J	250 U	250 U	250 U	12,000,000
MFAPES154	10/28/98	1	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES155	10/26/98	2	0 - 6	MK	250 U	260 U	442	391	250 U	250 U	250 U	12,700,000 *

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES127	10/20/98	203	Sidewall	MK	N/A	91,100	140 B	12,400 N	14,200 B	160 *	N/A	N/A
MFAPES127FD	10/20/98	203	Sidewall	MK	N/A	94,900	150 B	17,100 N	14,000 B	30 *B	N/A	N/A
MFAPES128	10/20/98	203	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES129	10/20/98	45	0 - 6	MK	N/A	60,200	120 B	16,600	11,400 B	40 *U	N/A	N/A
MFAPES130	10/20/98	45	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES131	10/20/98	48	0 - 6	MK	N/A	85,600	120 B	14,000	12,100 B	40 *U	N/A	N/A
MFAPES132	10/20/98	48	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES133	10/20/98	50	0 - 6	MK	N/A	39,000	90 B	16,200	10,300 B	40 *U	N/A	N/A
MFAPES134	10/20/98	50	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES135	10/20/98	52	0 - 6	MK	N/A	38,200	100 B	19,100	13,400 B	60 *	N/A	N/A
MFAPES136	10/20/98	52	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES137	10/22/98	54	0 - 6	MK	N/A	76,900	40 U	18,600 N	15,100 B	50 *	N/A	N/A
MFAPES138	10/22/98	54	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES139	10/22/98	56	0 - 6	MK	N/A	53,800	40 U	14,700 N	11,000 B	40 *	N/A	N/A
MFAPES140	10/22/98	56	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES141	10/22/98	49	0 - 6	MK	N/A	98,500	50 B	11,300 N	15,100 B	60 *	N/A	N/A
MFAPES142	10/22/98	49	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES143	10/22/98	43	0 - 6	MK	N/A	48,600	40 U	12,200 N	10,900 B	50 *	N/A	N/A
MFAPES144	10/22/98	43	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES145	10/23/98	166	Sidewall	MK	N/A	39,300	110 B	21,400 N*	14,100 EB	100	N/A	N/A
MFAPES146	10/26/98	166	Sidewall	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES147	10/26/98	143	0 - 6	MK	N/A	52,400	120 B	14,500 N*	8,800 EB	90	N/A	N/A
MFAPES147FD	10/26/98	143	0 - 6	MK	N/A	54,100	80 B	13,200 N*	11,800 EB	70	N/A	N/A
MFAPES148	10/26/98	143	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES149	10/28/98	15	0 - 6	MK	N/A	60,000	350 B	20,000	15,600 NB	40	N/A	N/A
MFAPES150	10/28/98	15	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES151	10/28/98	142	0 - 6	MK	N/A	60,800	280 B	16,100	12,900 NB	20 U	N/A	N/A
MFAPES152	10/28/98	142	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES153	10/28/98	1	0 - 6	MK	N/A	77,500	340 B	18,600	14,000 NB	20 U	N/A	N/A
MFAPES154	10/28/98	1	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES155	10/26/98	2	0 - 6	MK	N/A	79,100	110 B	18,700 N*	14,100 EB	40 U	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES156	10/26/98	2	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES157	10/27/98	66	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES158	10/27/98	66	6 - 6	MK	6 U	6 U	6 U	26	6 U	6 U	N/A
MFAPES159	10/27/98	183	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES160	10/27/98	183	6 - 6	MK	7 U	7 U	50	38	7	7 U	N/A
MFAPES161	10/27/98	51	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES162	10/27/98	51	6 - 6	MK	6 U	6 U	6 U	16	6 U	6 U	N/A
MFAPES163	10/27/98	67	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES164	10/27/98	67	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES165	10/29/98	53	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES166	10/29/98	53	6 - 6	MK	5 U	5 U	5 U	9	5 U	5 U	N/A
MFAPES167	10/29/98	55	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES167FD	10/29/98	55	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES168	10/29/98	55	6 - 6	MK	5 U	5 U	5 U	8	5 U	5 U	N/A
MFAPES169	10/29/98	57	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES170	10/29/98	57	6 - 6	MK	5 U	5 U	5 U	8	5 U	5 U	N/A
MFAPES171	11/11/98	65	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES172	11/11/98	65	6 - 6	MK	5 U	5 U	68	5 U	5 U	5 U	N/A
MFAPES173	11/11/98	64	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES174	11/11/98	64	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES175	11/11/98	10	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES176	11/11/98	10	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES177	11/11/98	11	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES178	11/11/98	11	6 - 6	MK	5 U	5 U	17	5 U	5 U	5 U	N/A
MFAPES179	11/11/98	62	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES180	11/11/98	62	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES181	11/12/98	12	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES182	11/12/98	12	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES183	11/12/98	63	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES184	11/12/98	63	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES185	11/12/98	149	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES156	10/26/98	2	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES157	10/27/98	66	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES158	10/27/98	66	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES159	10/27/98	183	0 - 6	MK	1,880 J	248 U	238 U	2,680	238 U	619 U	238 U
MFAPES160	10/27/98	183	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES161	10/27/98	51	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES162	10/27/98	51	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES163	10/27/98	67	0 - 6	MK	4,090	260 U	250 U	***16,000***	250 U	650 U	250 U
MFAPES164	10/27/98	67	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES165	10/29/98	53	0 - 6	MK	2,200	260 U	250 U	***6,110***	250 U	650 U	358
MFAPES166	10/29/98	53	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES167	10/29/98	55	0 - 6	MK	2,100 U	248 U	238 U	952 U	238 U	619 U	238 U
MFAPES167FD	10/29/98	55	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES168	10/29/98	55	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES169	10/29/98	57	0 - 6	MK	2,380	248 U	238 U	***5,390***	238 U	619 U	89.200 J
MFAPES170	10/29/98	57	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES171	11/11/98	65	0 - 6	MK	7,430	260 U	250 U	**56,200 D**	250 U	650 U	8,420
MFAPES172	11/11/98	65	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES173	11/11/98	64	0 - 6	MK	5,910	248 U	238 U	**47,300 D**	238 U	619 U	4,330
MFAPES174	11/11/98	64	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES175	11/11/98	10	0 - 6	MK	2,480	260 U	250 U	**20,500**	250 U	650 U	11,000
MFAPES176	11/11/98	10	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES177	11/11/98	11	0 - 6	MK	7,040	260 U	250 U	**40,000 D**	250 U	650 U	7,000
MFAPES178	11/11/98	11	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES179	11/11/98	62	0 - 6	MK	13,600	260 U	250 U	**64,000 D**	250 U	650 U	***21,600 D***
MFAPES180	11/11/98	62	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES181	11/12/98	12	0 - 6	MK	44,100	248 U	238 U	**246,000 D**	238 U	619 U	4,910
MFAPES182	11/12/98	12	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES183	11/12/98	63	0 - 6	MK	15,800	260 U	250 U	**115,000 D**	250 U	650 U	12,600
MFAPES184	11/12/98	63	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES185	11/12/98	149	0 - 6	MK	21,800	260 U	250 U	**98,700 D**	250 U	650 U	685

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES156	10/26/98	2	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES157	10/27/98	66	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,970,000
MFAPES158	10/27/98	66	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES159	10/27/98	183	0 - 6	MK	238 U	248 U	238 U	238 U	238 U	238 U	238 U	14,200,000
MFAPES160	10/27/98	183	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES161	10/27/98	51	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,430,000
MFAPES162	10/27/98	51	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES163	10/27/98	67	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	9,790,000
MFAPES164	10/27/98	67	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES165	10/29/98	53	0 - 6	MK	250 U	260 U	142 J	130 J	250 U	250 U	250 U	11,100,000 *
MFAPES166	10/29/98	53	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES167	10/29/98	55	0 - 6	MK	238 U	248 U	238 U	238 U	238 U	238 U	238 U	13,800,000 *
MFAPES167FD	10/29/98	55	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,900,000 *
MFAPES168	10/29/98	55	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES169	10/29/98	57	0 - 6	MK	238 U	248 U	238 U	101 J	238 U	238 U	238 U	11,500,000 *
MFAPES170	10/29/98	57	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES171	11/11/98	65	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,550,000
MFAPES172	11/11/98	65	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES173	11/11/98	64	0 - 6	MK	238 U	248 U	664	1,120	238 U	238 U	238 U	7,220,000
MFAPES174	11/11/98	64	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES175	11/11/98	10	0 - 6	MK	250 U	260 U	708	250 U	250 U	250 U	250 U	9,010,000
MFAPES176	11/11/98	10	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES177	11/11/98	11	0 - 6	MK	250 U	260 U	863	1,400	250 U	250 U	250 U	9,670,000
MFAPES178	11/11/98	11	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES179	11/11/98	62	0 - 6	MK	250 U	260 U	250 U	3,120	250 U	250 U	250 U	10,400,000
MFAPES180	11/11/98	62	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES181	11/12/98	12	0 - 6	MK	238 U	248 U	524	4,760 U	238 U	238 U	238 U	13,800,000
MFAPES182	11/12/98	12	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES183	11/12/98	63	0 - 6	MK	250 U	260 U	250 U	2,110 DPJ	744	250 U	250 U	8,820,000
MFAPES184	11/12/98	63	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES185	11/12/98	149	0 - 6	MK	250 U	260 U	250 U	773 DPJ	250 U	250 U	250 U	7,420,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES156	10/26/98	2	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES157	10/27/98	66	0 - 6	MK	N/A	67,500	190 B	15,500	15,000 B	30 *B	N/A	N/A
MFAPES158	10/27/98	66	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES159	10/27/98	183	0 - 6	MK	N/A	66,900	170 B	16,400	15,300 B	60 *	N/A	N/A
MFAPES160	10/27/98	183	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES161	10/27/98	51	0 - 6	MK	N/A	67,800	150 B	11,900	10,700 B	20 *B	N/A	N/A
MFAPES162	10/27/98	51	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES163	10/27/98	67	0 - 6	MK	N/A	55,700	220 B	13,800	13,600 B	30 *B	N/A	N/A
MFAPES164	10/27/98	67	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES165	10/29/98	53	0 - 6	MK	N/A	63,100 N*	210 N*B	14,200 N*	11,200 NB	20 U	N/A	N/A
MFAPES166	10/29/98	53	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES167	10/29/98	55	0 - 6	MK	N/A	78,100 N*	240 N*B	17,700 N*	12,400 NB	20 U	N/A	N/A
MFAPES167FD	10/29/98	55	0 - 6	MK	N/A	62,200 N*	240 N*B	18,300 N*	14,000 NB	20 U	N/A	N/A
MFAPES168	10/29/98	55	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES169	10/29/98	57	0 - 6	MK	N/A	65,900 N*	220 N*B	12,900 N*	12,700 NB	20 U	N/A	N/A
MFAPES170	10/29/98	57	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES171	11/11/98	65	0 - 6	MK	N/A	59,600	60 B	12,000 N	10,500 B	60	N/A	N/A
MFAPES172	11/11/98	65	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES173	11/11/98	64	0 - 6	MK	N/A	44,300	150 B	10,100 N	8,700 B	90	N/A	N/A
MFAPES174	11/11/98	64	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES175	11/11/98	10	0 - 6	MK	N/A	49,300	60 B	12,300 N	8,600 B	60	N/A	N/A
MFAPES176	11/11/98	10	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES177	11/11/98	11	0 - 6	MK	N/A	53,700	50 B	13,300 N	10,000 B	60	N/A	N/A
MFAPES178	11/11/98	11	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES179	11/11/98	62	0 - 6	MK	N/A	60,000	40 B	13,400 N	12,600 B	40	N/A	N/A
MFAPES180	11/11/98	62	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES181	11/12/98	12	0 - 6	MK	N/A	105,000 *	230 *B	14,200 N*	15,200 *	1,600	N/A	N/A
MFAPES182	11/12/98	12	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES183	11/12/98	63	0 - 6	MK	N/A	59,500 *	330 *B	15,700 N*	12,900 *	1,000	N/A	N/A
MFAPES184	11/12/98	63	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES185	11/12/98	149	0 - 6	MK	N/A	66,200 *	200 *B	16,500 N*	13,800 *	40	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES186	11/12/98	149	6 - 6	MK	6 U	6 U	58	6 U	6 U	6 U	N/A
MFAPES187	11/12/98	153	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES187FD	11/12/98	153	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES188	11/12/98	153	6 - 6	MK	6 U	6 U	110	6 U	6 U	6 U	N/A
MFAPES189	11/12/98	150	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES190	11/12/98	150	6 - 6	MK	6 U	6 U	26	6 U	6 U	6 U	N/A
MFAPES191	11/13/98	182	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES192	11/16/98	182	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES193	11/13/98	151	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES194	11/16/98	151	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES195	11/13/98	70	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES196	11/16/98	70	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES197	11/13/98	58	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES198	11/16/98	58	6 - 6	MK	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES199	11/13/98	59	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES200	11/16/98	59	6 - 6	MK	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES201	11/19/98	75	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES202	11/19/98	75	6 - 6	MK	6 U	6 U	22	8 B	6 U	6 U	N/A
MFAPES203	11/19/98	76	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES204	11/19/98	76	6 - 6	MK	5 U	5 U	5 U	6 B	5 U	5 U	N/A
MFAPES205	11/19/98	77	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES206	11/19/98	77	6 - 6	MK	5 U	5 U	20	6 B	5 U	5 U	N/A
MFAPES207	11/25/98	88	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES207FD	11/25/98	88	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES208	11/30/98	88	6 - 6	MK	5 U	5 U	18	14	5 U	5 U	N/A
MFAPES209	12/09/98	74	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES210	12/09/98	74	6 - 6	MK	5 U	5 U	15 B	5 U	5 U	5 U	N/A
MFAPES211	12/09/98	73	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES212	12/09/98	73	6 - 6	MK	5 U	5 U	8 B	5 U	5 U	5 U	N/A
MFAPES213	12/09/98	72	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES214	12/09/98	72	6 - 6	MK	5 U	5 U	15 B	5 U	5 U	5 U	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES186	11/12/98	149	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES187	11/12/98	153	0 - 6	MK	2,660	260 U	250 U	***4,220***	250 U	650 U	250 U
MFAPES187FD	11/12/98	153	0 - 6	MK	2,490	260 U	250 U	***4,480***	250 U	650 U	250 U
MFAPES188	11/12/98	153	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES189	11/12/98	150	0 - 6	MK	42,000	260 U	250 U	**215,000 D**	250 U	650 U	640
MFAPES190	11/12/98	150	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES191	11/13/98	182	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES192	11/16/98	182	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES193	11/13/98	151	0 - 6	MK	2,830	260 U	250 U	***16,100***	250 U	650 U	250 U
MFAPES194	11/16/98	151	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES195	11/13/98	70	0 - 6	MK	364 J	260 U	250 U	3,510	250 U	650 U	250 U
MFAPES196	11/16/98	70	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES197	11/13/98	58	0 - 6	MK	87,900 DJ	260 U	250 U	**752,000 D**	250 U	650 U	**262,000 D**
MFAPES198	11/16/98	58	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES199	11/13/98	59	0 - 6	MK	22,300	260 U	250 U	**144,000 D**	250 U	650 U	2,760
MFAPES200	11/16/98	59	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES201	11/19/98	75	0 - 6	MK	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES202	11/19/98	75	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES203	11/19/98	76	0 - 6	MK	601 J	260 U	250 U	632 J	250 U	650 U	1,220
MFAPES204	11/19/98	76	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES205	11/19/98	77	0 - 6	MK	1,280 J	260 U	250 U	***4,950***	250 U	650 U	250 U
MFAPES206	11/19/98	77	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES207	11/25/98	88	0 - 6	MK	2,620	260 U	250 U	2,620	250 U	650 U	250 U
MFAPES207FD	11/25/98	88	0 - 6	MK	2,300	260 U	250 U	2,500	250 U	650 U	236 J
MFAPES208	11/30/98	88	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES209	12/09/98	74	0 - 6	MK	1,240 J	260 U	250 U	3,380	250 U	650 U	250 U
MFAPES210	12/09/98	74	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES211	12/09/98	73	0 - 6	MK	1,150 J	260 U	250 U	2,640	250 U	650 U	250 U
MFAPES212	12/09/98	73	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES213	12/09/98	72	0 - 6	MK	5,800	260 U	250 U	**40,300 D**	922	650 U	***21,400 D***
MFAPES214	12/09/98	72	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES186	11/12/98	149	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES187	11/12/98	153	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10,400,000
MFAPES187FD	11/12/98	153	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	N/A
MFAPES188	11/12/98	153	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES189	11/12/98	150	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,400,000
MFAPES190	11/12/98	150	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES191	11/13/98	182	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,100,000
MFAPES192	11/16/98	182	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES193	11/13/98	151	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,600,000
MFAPES194	11/16/98	151	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES195	11/13/98	70	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,900,000
MFAPES196	11/16/98	70	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES197	11/13/98	58	0 - 6	MK	242 PJ	1,410	250 U	250 U	939 P	250 U	250 U	14,400,000
MFAPES198	11/16/98	58	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES199	11/13/98	59	0 - 6	MK	250 U	260 U	1,820	1,720	250 U	250 U	250 U	19,200,000
MFAPES200	11/16/98	59	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES201	11/19/98	75	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,200,000
MFAPES202	11/19/98	75	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES203	11/19/98	76	0 - 6	MK	250 U	260 U	368	240 J	250 U	250 U	250 U	14,000,000
MFAPES204	11/19/98	76	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES205	11/19/98	77	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,000,000
MFAPES206	11/19/98	77	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES207	11/25/98	88	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,900,000
MFAPES207FD	11/25/98	88	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	10,400,000
MFAPES208	11/30/98	88	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES209	12/09/98	74	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,900,000
MFAPES210	12/09/98	74	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES211	12/09/98	73	0 - 6	MK	250 U	260 U	250 U	250 U	250 U	250 U	250 U	8,780,000
MFAPES212	12/09/98	73	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES213	12/09/98	72	0 - 6	MK	250 U	260 U	250 U	1,000	250 U	250 U	250 U	10,500,000
MFAPES214	12/09/98	72	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES186	11/12/98	149	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES187	11/12/98	153	0 - 6	MK	N/A	73,600 *	170 *B	15,900 N*	15,400 *	40	N/A	N/A
MFAPES187FD	11/12/98	153	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES188	11/12/98	153	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES189	11/12/98	150	0 - 6	MK	N/A	57,700 *	290 *B	11,800 N*	13,700 *	60	N/A	N/A
MFAPES190	11/12/98	150	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES191	11/13/98	182	0 - 6	MK	N/A	66,800	110 B	13,100	11,500 B	40 U	N/A	N/A
MFAPES192	11/16/98	182	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES193	11/13/98	151	0 - 6	MK	N/A	75,600	130 B	14,700	13,700 B	40 U	N/A	N/A
MFAPES194	11/16/98	151	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES195	11/13/98	70	0 - 6	MK	N/A	88,200	160 B	17,500	11,900 B	40 U	N/A	N/A
MFAPES196	11/16/98	70	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES197	11/13/98	58	0 - 6	MK	N/A	73,300	130 B	18,300	15,200 B	40 U	N/A	N/A
MFAPES198	11/16/98	58	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES199	11/13/98	59	0 - 6	MK	N/A	66,700	110 B	24,300	10,000 B	40 U	N/A	N/A
MFAPES200	11/16/98	59	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES201	11/19/98	75	0 - 6	MK	N/A	77,400	40 U	19,200	17,500 B	50	N/A	N/A
MFAPES202	11/19/98	75	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES203	11/19/98	76	0 - 6	MK	N/A	79,800	30 U	16,400	12,600 B	40	N/A	N/A
MFAPES204	11/19/98	76	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES205	11/19/98	77	0 - 6	MK	N/A	67,900	30 U	14,500	11,700 B	60	N/A	N/A
MFAPES206	11/19/98	77	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES207	11/25/98	88	0 - 6	MK	N/A	69,600	110 B	16,400	16,300 N	60	N/A	N/A
MFAPES207FD	11/25/98	88	0 - 6	MK	N/A	84,800	90 B	13,500	11,800 N	70	N/A	N/A
MFAPES208	11/30/98	88	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES209	12/09/98	74	0 - 6	MK	N/A	82,700	200 B	15,000	16,300 B	90	N/A	N/A
MFAPES210	12/09/98	74	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES211	12/09/98	73	0 - 6	MK	N/A	86,100	160 B	12,000	13,900 B	50	N/A	N/A
MFAPES212	12/09/98	73	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES213	12/09/98	72	0 - 6	MK	N/A	147,000	220 B	14,300	14,200 B	50	N/A	N/A
MFAPES214	12/09/98	72	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES215	12/09/98	71	0 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES216	12/09/98	71	6 - 6	MK	6 U	6 U	6 U	2 J	6 U	6 U	N/A
MFAPES217	04/08/99	117	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES218	04/08/99	117	6	TOLTEST	6 U	6 U	6 U	9 B	6 U	6 U	N/A
MFAPES219	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES220	04/08/99	117	sidewall	TOLTEST	6 U	6 U	6 U	10 B	6 U	6 U	N/A
MFAPES221	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES222	04/08/99	117	sidewall	TOLTEST	5 U	5 U	5 U	10 B	5 U	5 U	N/A
MFAPES223	04/28/99	106	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES224	04/28/99	106	6	TOLTEST	11 U	11 U	11 U	5 J	5 U	5 U	N/A
MFAPES225	04/28/99	107	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES226	04/28/99	107	6	TOLTEST	9 U	9 U	40	3 J	5 U	5 U	N/A
MFAPES227	04/28/99	108	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	192 U
MFAPES228	04/28/99	108	6	TOLTEST	9 U	9 U	9 U	4 J	5 U	5 U	N/A
MFAPES229	04/28/99	109	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	208 U
MFAPES229FD	04/28/99	109	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES230	04/28/99	109	6	TOLTEST	10 U	10 U	10 U	4 J	5 U	5 U	N/A
MFAPES231	04/29/99	105	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES232	04/29/99	105	6	TOLTEST	9 U	9 U	9 U	4 J	5 U	5 U	N/A
MFAPES233	04/29/99	104	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	227 U
MFAPES234	04/29/99	104	6	TOLTEST	9 U	9 U	9 U	4 J	4 U	4 U	N/A
MFAPES235	04/29/99	103	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	227 U
MFAPES236	04/29/99	103	6	TOLTEST	8 U	8 U	8 U	3 J	4 U	4 U	N/A
MFAPES237	05/21/99	100	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES237-FD	05/21/99	100	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES239	05/21/99	101	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES240	05/21/99	101	6	TOLTEST	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES241	05/21/99	112	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES242	05/21/99	112	6	TOLTEST	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES243	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES215	12/09/98	71	0 - 6	MK	4,200	260 U	250 U	***8,380***	250 U	650 U	***19,400 D***
MFAPES216	12/09/98	71	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES217	04/08/99	117	0-6	TOLTEST	1,030 J	260 U	250 U	1350	250 U	650 U	482
MFAPES218	04/08/99	117	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES219	04/08/99	117	sidewall	TOLTEST	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES220	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES221	04/08/99	117	sidewall	TOLTEST	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES222	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES223	04/28/99	106	0-6	TOLTEST	3,390	248 U	238 U	**19,200**	804 P	619 U	4,020
MFAPES224	04/28/99	106	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES225	04/28/99	107	0-6	TOLTEST	1,760 U	208 U	200 U	3,260	256 P	520 U	200 U
MFAPES226	04/28/99	107	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES227	04/28/99	108	0-6	TOLTEST	1,690 U	200 U	192 U	3,530	192 U	500 U	267
MFAPES228	04/28/99	108	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES229	04/28/99	109	0-6	TOLTEST	1,830 U	217 U	208 U	***6,320***	208 U	542 U	618
MFAPES229FD	04/28/99	109	0-6	TOLTEST	2,100 U	248 U	238 U	***6,280***	238 U	619 U	296
MFAPES230	04/28/99	109	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES231	04/29/99	105	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES232	04/29/99	105	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES233	04/29/99	104	0-6	TOLTEST	2,950	236 U	250 U	***8,950***	227 U	591 U	227 U
MFAPES234	04/29/99	104	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES235	04/29/99	103	0-6	TOLTEST	4,000	236 U	250 U	**19,400**	227 U	591 U	1,680
MFAPES236	04/29/99	103	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES237	05/21/99	100	0-6	TOLTEST	2,100 U	248 U	250 U	952 U	238 U	619 U	238 U
MFAPES237-FD	05/21/99	100	0-6	TOLTEST	2,100 U	248 U	250 U	952 U	238 U	619 U	238 U
MFAPES239	05/21/99	101	0-6	TOLTEST	2,170	248 U	238 U	***8,640***	238 U	619 U	1350
MFAPES240	05/21/99	101	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES241	05/21/99	112	0-6	TOLTEST	2,100 U	248 U	238 U	2,490	238 U	619 U	238 U
MFAPES242	05/21/99	112	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES243	05/21/99	112	sidewall	TOLTEST	2,100 U	248 U	238 U	1,770 P	***5,260***	619 U	***27,800 D***

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES215	12/09/98	71	0 - 6	MK	250 U	260 U	250 U	871 P	250 U	250 U	250 U	10,500,000
MFAPES216	12/09/98	71	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES217	04/08/99	117	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,500,000
MFAPES218	04/08/99	117	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES219	04/08/99	117	sidewall	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,200,000
MFAPES220	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES221	04/08/99	117	sidewall	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	12,600,000
MFAPES222	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES223	04/28/99	106	0-6	TOLTEST	238 U	248 U	1230	707	238 U	238 U	238 U	9,660,000
MFAPES224	04/28/99	106	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES225	04/28/99	107	0-6	TOLTEST	200 U	208 U	200 U	200 U	200 U	200 U	200 U	12,400,000
MFAPES226	04/28/99	107	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES227	04/28/99	108	0-6	TOLTEST	192 U	200 U	192 U	192 U	192 U	192 U	192 U	12,200,000
MFAPES228	04/28/99	108	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES229	04/28/99	109	0-6	TOLTEST	208 U	217 U	460	234	208 U	208 U	208 U	12,100,000
MFAPES229FD	04/28/99	109	0-6	TOLTEST	238 U	248 U	502	246	238 U	238 U	238 U	11,600,000
MFAPES230	04/28/99	109	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES231	04/29/99	105	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	9,560,000
MFAPES232	04/29/99	105	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES233	04/29/99	104	0-6	TOLTEST	227 U	236 U	227 U	227 U	227 U	227 U	227 U	8,890,000
MFAPES234	04/29/99	104	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES235	04/29/99	103	0-6	TOLTEST	227 U	236 U	209 J	383	227 U	227 U	227 U	9,560,000
MFAPES236	04/29/99	103	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES237	05/21/99	100	0-6	TOLTEST	238 U	248 U	238 U	238 U	238 U	238 U	238 U	16,400,000
MFAPES237-FD	05/21/99	100	0-6	TOLTEST	238 U	248 U	238 U	238 U	238 U	238 U	238 U	14,300,000
MFAPES239	05/21/99	101	0-6	TOLTEST	238 U	248 U	546	1620	238 U	238 U	238 U	14,600,000
MFAPES240	05/21/99	101	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES241	05/21/99	112	0-6	TOLTEST	238 U	248 U	238 U	238 U	238 U	238 U	238 U	13,200,000
MFAPES242	05/21/99	112	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES243	05/21/99	112	sidewall	TOLTEST	238 U	248 U	1,280	1,590	238 U	238 U	238 U	5,300,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES215	12/09/98	71	0 - 6	MK	N/A	102,000	220 B	14,600	15,200 B	40	N/A	N/A
MFAPES216	12/09/98	71	6 - 6	MK	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES217	04/08/99	117	0-6	TOLTEST	**8,300**	71,600	100 B	16,000	12,800	20 U	550 U	250 U
MFAPES218	04/08/99	117	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES219	04/08/99	117	sidewall	TOLTEST	**7,400**	66,200	40 U	15,500	12,200	20 U	540 U	240 U
MFAPES220	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES221	04/08/99	117	sidewall	TOLTEST	**9,300**	74,800	40 U	16,200	13,200	20 U	540 U	240 U
MFAPES222	04/08/99	117	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES223	04/28/99	106	0-6	TOLTEST	**8,200**	61,200	40 U	13,100	12,800 B	50	570 B	140 NU
MFAPES224	04/28/99	106	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES225	04/28/99	107	0-6	TOLTEST	**9,700**	63,200	40 U	17,100	13,200 B	60	910	150 NU
MFAPES226	04/28/99	107	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES227	04/28/99	108	0-6	TOLTEST	**10,500**	77,200	40 U	15,500	13,000 B	230	820	140 NU
MFAPES228	04/28/99	108	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES229	04/28/99	109	0-6	TOLTEST	**8,500**	69,600	40 B	14,500	13,900 B	60	940	150 NU
MFAPES229FD	04/28/99	109	0-6	TOLTEST	**10,500**	66,900	40 U	16,000	14,800 B	70	580 U	150 NU
MFAPES230	04/28/99	109	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES231	04/29/99	105	0-6	TOLTEST	**8,600**	60,200	40 U	12,900	12,500 B	30 B	590 U	150 U
MFAPES232	04/29/99	105	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES233	04/29/99	104	0-6	TOLTEST	**8,100**	57,200	950 U	13,100	11,700 B	40	580 U	150 U
MFAPES234	04/29/99	104	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES235	04/29/99	103	0-6	TOLTEST	**8,100**	69,000	950 U	12,100	11,900 B	40	570 U	150 U
MFAPES236	04/29/99	103	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES237	05/21/99	100	0-6	TOLTEST	**13,000**	88,000	90 B	19,000	14,000	30 B	440 U	150 U
MFAPES237-FD	05/21/99	100	0-6	TOLTEST	**8,000**	84,000	120 B	17,000	12,000	30 B	400 U	150 U
MFAPES239	05/21/99	101	0-6	TOLTEST	**9,000**	103,000	110 B	17,000	14,000	20 U	400 U	150 U
MFAPES240	05/21/99	101	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES241	05/21/99	112	0-6	TOLTEST	**7,000**	50,000	100 B	16,000	9,000	20 B	390 U	140 U
MFAPES242	05/21/99	112	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES243	05/21/99	112	sidewall	TOLTEST	**5,000**	27,000	190 B	9,000	7,000	30 B	410 U	150 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES244	05/21/99	112	sidewall	TOLTEST	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES245	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES246	05/21/99	112	sidewall	TOLTEST	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES247	05/21/99	102	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES248	05/21/99	102	6	TOLTEST	6 U	6 U	15	6 U	6 U	6 U	N/A
MFAPES249	05/21/99	114	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES250	05/21/99	114	6	TOLTEST	6 U	6 U	21	6 U	6 U	6 U	N/A
MFAPES251	05/21/99	185	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES252	05/21/99	185	6	TOLTEST	6 U	6 U	6	6 U	6 U	6 U	N/A
MFAPES253	05/26/99	100	6	TOLTEST	6 U	6 U	15	6 U	6 U	6 U	N/A
MFAPES254	05/26/99	99	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	245 U
MFAPES254FD	05/26/99	99	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	232 U
MFAPES255	05/26/99	99	6	TOLTEST	6 U	6 U	13	6 U	6 U	6 U	N/A
MFAPES256	05/26/99	98	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	237 U
MFAPES257	05/26/99	98	6	TOLTEST	5 U	5 U	16	5 U	5 U	5 U	N/A
MFAPES258	05/26/99	97	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES259	05/26/99	97	6	TOLTEST	6 U	6 U	47	6 U	6 U	6 U	N/A
MFAPES260	05/26/99	96	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	239 U
MFAPES261	05/26/99	96	6	TOLTEST	6 U	6 U	69	6 U	6 U	6 U	N/A
MFAPES262	06/03/99	96	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	208 U
MFAPES263	06/03/99	96	6	TOLTEST	6 U	6 U	6 U	35	6 U	6 U	N/A
MFAPES264	06/07/99	96	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES265	06/15/99	90	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES265FD	06/15/99	90	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES266	06/15/99	90	6	TOLTEST	6 U	6 U	6 U	6 U	6 U	6 U	N/A
MFAPES267	06/15/99	90	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES268	06/15/99	90	sidewall	TOLTEST	5 U	5 U	3 J	5 U	5 U	5 U	N/A
MFAPES269	06/17/99	78	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES270	06/21/99	78	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES270FD	06/21/99	78	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES244	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES245	05/21/99	112	sidewall	TOLTEST	4,120	248 U	238 U	952 U	238 U	619 U	238 U
MFAPES246	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES247	05/21/99	102	0-6	TOLTEST	1,220 J	248 U	238 U	***5,390***	238 U	619 U	1,180
MFAPES248	05/21/99	102	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES249	05/21/99	114	0-6	TOLTEST	4,170	248 U	250 U	***5,440***	238 U	619 U	5,020
MFAPES250	05/21/99	114	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES251	05/21/99	185	0-6	TOLTEST	6,660	248 U	250 U	3,840	238 U	619 U	4,600
MFAPES252	05/21/99	185	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES253	05/26/99	100	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES254	05/26/99	99	0-6	TOLTEST	2,160 U	255 U	245 U	***6,730***	471 P	637 U	802
MFAPES254FD	05/26/99	99	0-6	TOLTEST	2,050 U	242 U	232 U	***7,060***	579 P	605 U	987
MFAPES255	05/26/99	99	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES256	05/26/99	98	0-6	TOLTEST	2,080 U	246 U	237 U	948 U	237 U	616 U	2,610
MFAPES257	05/26/99	98	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES258	05/26/99	97	0-6	TOLTEST	2,100 U	248 U	238 U	952 U	238 U	619 U	241
MFAPES259	05/26/99	97	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES260	05/26/99	96	0-6	TOLTEST	4,250	249 U	239 U	**53,600 D**	239 U	622 U	***18,300 D***
MFAPES261	05/26/99	96	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES262	06/03/99	96	0-6	TOLTEST	1,830 U	217 U	208 U	833 U	208 U	542 U	208 U
MFAPES263	06/03/99	96	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES264	06/07/99	96	0-6	TOLTEST	2,270	260 U	250 U	***8,290***	250 U	650 U	250 U
MFAPES265	06/15/99	90	0-6	TOLTEST	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES265FD	06/15/99	90	0-6	TOLTEST	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES266	06/15/99	90	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES267	06/15/99	90	sidewall	TOLTEST	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES268	06/15/99	90	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES269	06/17/99	78	0-6	TOLTEST	2,150	248 U	250 U	***11,000***	238 U	619 U	238 U
MFAPES270	06/21/99	78	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES270FD	06/21/99	78	0-6	TOLTEST	2,200 U	260 U	250 U	827 J	250 U	650 U	250 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES244	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES245	05/21/99	112	sidewall	TOLTEST	238 U	248 U	238 U	238 U	238 U	238 U	238 U	10,200,000
MFAPES246	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES247	05/21/99	102	0-6	TOLTEST	238 U	248 U	312	400	238 U	238 U	238 U	13,800,000
MFAPES248	05/21/99	102	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES249	05/21/99	114	0-6	TOLTEST	238 U	248 U	331	352	238 U	238 U	238 U	12,200,000
MFAPES250	05/21/99	114	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES251	05/21/99	185	0-6	TOLTEST	238 U	248 U	238 U	238 U	238 U	238 U	238 U	15,000,000
MFAPES252	05/21/99	185	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES253	05/26/99	100	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES254	05/26/99	99	0-6	TOLTEST	245 U	255 U	385	526	245 U	245 U	245 U	11,400,000
MFAPES254FD	05/26/99	99	0-6	TOLTEST	232 U	242 U	416	604	232 U	232 U	232 U	13,800,000
MFAPES255	05/26/99	99	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES256	05/26/99	98	0-6	TOLTEST	237 U	246 U	237 U	237 U	237 U	237 U	237 U	12,700,000
MFAPES257	05/26/99	98	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES258	05/26/99	97	0-6	TOLTEST	238 U	248 U	238 U	238 U	238 U	238 U	238 U	13,900,000
MFAPES259	05/26/99	97	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES260	05/26/99	96	0-6	TOLTEST	239 U	249 U	570	628	239 U	239 U	239 U	12,200,000
MFAPES261	05/26/99	96	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES262	06/03/99	96	0-6	TOLTEST	208 U	217 U	208 U	208 U	208 U	208 U	208 U	7,250,000
MFAPES263	06/03/99	96	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES264	06/07/99	96	0-6	TOLTEST	250 U	260 U	196 J	539	250 U	250 U	250 U	12,400,000
MFAPES265	06/15/99	90	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	18,100,000 *
MFAPES265FD	06/15/99	90	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	17,700,000 *
MFAPES266	06/15/99	90	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES267	06/15/99	90	sidewall	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	13,700,000 *
MFAPES268	06/15/99	90	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES269	06/17/99	78	0-6	TOLTEST	238 U	248 U	238 U	225 J	238 U	238 U	238 U	10,700,000
MFAPES270	06/21/99	78	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	15,900,000
MFAPES270FD	06/21/99	78	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,300,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES244	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES245	05/21/99	112	sidewall	TOLTEST	7,000	88,000	170 B	12,000	13,000	20 B	400 U	150 U
MFAPES246	05/21/99	112	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES247	05/21/99	102	0-6	TOLTEST	8,000	104,000	100 B	17,000	14,000	30 B	400 U	150 U
MFAPES248	05/21/99	102	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES249	05/21/99	114	0-6	TOLTEST	8,000	89,000	120 B	15,000	14,000	20 U	400 U	140 U
MFAPES250	05/21/99	114	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES251	05/21/99	185	0-6	TOLTEST	10,000	64,000	100 B	19,000	13,000	40	440 U	150 U
MFAPES252	05/21/99	185	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES253	05/26/99	100	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES254	05/26/99	99	0-6	TOLTEST	7,900	78,800	60 B	15,100	13,300	80 N	540 U	240 U
MFAPES254FD	05/26/99	99	0-6	TOLTEST	8,700	83,900	70 B	17,000	13,800	70 N	530 U	240 U
MFAPES255	05/26/99	99	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES256	05/26/99	98	0-6	TOLTEST	7,800	81,700	40 B	16,700	12,700	200 N	540 U	240 U
MFAPES257	05/26/99	98	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES258	05/26/99	97	0-6	TOLTEST	7,000	62,800	40 U	17,500	11,700	90 N	560 U	250 U
MFAPES259	05/26/99	97	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES260	05/26/99	96	0-6	TOLTEST	10,000	66,200	50 B	16,400	13,400	70 N	550 U	250 U
MFAPES261	05/26/99	96	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES262	06/03/99	96	0-6	TOLTEST	5,000	35,000	90 B	10,000	8,000	30 B	420 U	150 U
MFAPES263	06/03/99	96	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES264	06/07/99	96	0-6	TOLTEST	11,000	65,000	100 B	17,000	18,000 B	80	380 U	140 U
MFAPES265	06/15/99	90	0-6	TOLTEST	14,000	70,000	40 U	22,000 N	20,000	70	1000 B	150 U
MFAPES265FD	06/15/99	90	0-6	TOLTEST	9,000	69,000	40 U	22,000 N	13,000	40 B	410 U	150 U
MFAPES266	06/15/99	90	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES267	06/15/99	90	sidewall	TOLTEST	9,000	100,000	30 U	18,000 N	14,000	30 B	380 U	140 U
MFAPES268	06/15/99	90	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES269	06/17/99	78	0-6	TOLTEST	8,000	105,000	40 U	14,000	16,000	20 U	400 U	140 U
MFAPES270	06/21/99	78	0-6	TOLTEST	8,000 N*	87,000 N*	40 NU	21,000 N	12,000 N*	30 B	400 N*U	150 U
MFAPES270FD	06/21/99	78	0-6	TOLTEST	6,000 N*	93,000 N*	40 NU	20,000 N	12,000 N*	30 B	400 N*U	150 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES271	06/21/99	78	6	TOLTEST	7 U	7 U	9	4 JB	7 U	7 U	N/A
MFAPES272	06/21/99	78	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES273	06/21/99	78	sidewall	TOLTEST	6 U	6 U	6 U	4 JB	6 U	6 U	N/A
MFAPES274	06/21/99	82	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES275	06/21/99	82	6	TOLTEST	5 U	5 U	10	5 JB	5 U	5 U	N/A
MFAPES276	06/21/99	83	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES277	06/21/99	83	6	TOLTEST	6 U	6 U	6 U	4 JB	6 U	6 U	N/A
MFAPES278	06/21/99	84	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES279	06/21/99	84	6	TOLTEST	6 U	6 U	6 U	5 JB	6 U	6 U	N/A
MFAPES280	06/21/99	92	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES281	06/21/99	92	6	TOLTEST	5 U	5 U	5 U	3 JB	5 U	5 U	N/A
MFAPES282	08/04/99	28	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES282FD	08/04/99	28	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES283	08/04/99	28	6	TOLTEST	5 U	5 U	5 U	5 U	5 U	5 U	N/A
MFAPES284	08/04/99	27	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	238 U
MFAPES285	08/04/99	27	6	TOLTEST	7 U	7 U	7 U	21 B	7 U	7 U	N/A
MFAPES286	08/04/99	31	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES287	08/04/99	31	6	TOLTEST	5 U	5 U	5 U	15 B	5 U	5 U	N/A
MFAPES288	08/09/99	32	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES288FD	08/09/99	32	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES289	08/09/99	32	6	TOLTEST	9 U	4 U	23 B	4 U	4 U	4 U	N/A
MFAPES290	08/10/99	30	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES290FD	08/10/99	30	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES291	08/10/99	30	6	TOLTEST	11 U	6 U	13 J	6 U	6 U	6 U	N/A
MFAPES292	08/11/99	130	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES293	08/11/99	130	6	TOLTEST	12 U	6 U	14 J	6 U	6 U	6 U	N/A
MFAPES294	08/11/99	39	0-6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	250 U
MFAPES295	08/11/99	39	6	TOLTEST	13 U	6 U	21 J	6 U	6 U	6 U	N/A
MFAPES296	08/27/99	60	0-7	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	1,000 U
MFAPES296FD	08/27/99	60	0-7	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	1,000 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES271	06/21/99	78	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES272	06/21/99	78	sidewall	TOLTEST	4,870	260 U	250 U	***12,900***	250 U	650 U	250 U
MFAPES273	06/21/99	78	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES274	06/21/99	82	0-6	TOLTEST	2,200 U	260 U	250 U	1,000 U	250 U	650 U	250 U
MFAPES275	06/21/99	82	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES276	06/21/99	83	0-6	TOLTEST	2,200 U	260 U	250 U	***7,290***	250 U	650 U	250 U
MFAPES277	06/21/99	83	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES278	06/21/99	84	0-6	TOLTEST	2,200 U	260 U	250 U	2,920	250 U	650 U	250 U
MFAPES279	06/21/99	84	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES280	06/21/99	92	0-6	TOLTEST	1,700 J	260 U	250 U	***6,330***	250 U	650 U	2,730
MFAPES281	06/21/99	92	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES282	08/04/99	28	0-6	TOLTEST	24,400	260 U	250 U	**199,000 D**	250 U	650 U	1,140
MFAPES282FD	08/04/99	28	0-6	TOLTEST	55,400	260 U	250 U	**353,000 D**	250 U	650 U	2,200
MFAPES283	08/04/99	28	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES284	08/04/99	27	0-6	TOLTEST	38,500	248 U	250 U	**266,000 D**	238 U	619 U	8,260
MFAPES285	08/04/99	27	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES286	08/04/99	31	0-6	TOLTEST	4,380	260 U	250 U	***13,700***	250 U	650 U	750
MFAPES287	08/04/99	31	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES288	08/09/99	32	0-6	TOLTEST	1,500	250 U	*500 U*	3,100	250 U	250 U	700
MFAPES288FD	08/09/99	32	0-6	TOLTEST	1,300	250 U	*500 U*	2,100	250 U	250 U	790
MFAPES289	08/09/99	32	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES290	08/10/99	30	0-6	TOLTEST	570	250 U	*500 U*	2,700	250 U	250 U	250 U
MFAPES290FD	08/10/99	30	0-6	TOLTEST	500	250 U	*500 U*	2,500	250 U	250 U	250 U
MFAPES291	08/10/99	30	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES292	08/11/99	130	0-6	TOLTEST	1,800	250 U	*500 U*	***5,800***	250 U	250 U	250 U
MFAPES293	08/11/99	130	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES294	08/11/99	39	0-6	TOLTEST	5,100	250 U	*500 U*	***6,700***	250 U	250 U	250 U
MFAPES295	08/11/99	39	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES296	08/27/99	60	0-7	TOLTEST	1,210	1,000 U	*2,500 U*	963 J	1,000 U	1,000 U	1,000 U
MFAPES296FD	08/27/99	60	0-7	TOLTEST	2,720	1,000 U	*2,500 U*	2,020	1,000 U	1,000 U	1,000 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES271	06/21/99	78	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES272	06/21/99	78	sidewall	TOLTEST	250 U	260 U	250 U	1,120 P	250 U	250 U	250 U	11,900,000
MFAPES273	06/21/99	78	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES274	06/21/99	82	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,600,000
MFAPES275	06/21/99	82	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES276	06/21/99	83	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	11,400,000
MFAPES277	06/21/99	83	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES278	06/21/99	84	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,900,000
MFAPES279	06/21/99	84	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES280	06/21/99	92	0-6	TOLTEST	250 U	260 U	250 U	250 U	250 U	250 U	250 U	15,500,000
MFAPES281	06/21/99	92	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES282	08/04/99	28	0-6	TOLTEST	250 U	260 U	368	1,340 P	250 U	250 U	250 U	10,800,000
MFAPES282FD	08/04/99	28	0-6	TOLTEST	250 U	260 U	586	1,850 P	250 U	250 U	250 U	11,600,000
MFAPES283	08/04/99	28	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES284	08/04/99	27	0-6	TOLTEST	238 U	248 U	1,390	2,570	238 U	238 U	238 U	9,840,000
MFAPES285	08/04/99	27	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES286	08/04/99	31	0-6	TOLTEST	250 U	260 U	692 P	464	250 U	250 U	250 U	11,500,000
MFAPES287	08/04/99	31	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES288	08/09/99	32	0-6	TOLTEST	250 U	250 U	98 J	180 J	74 J	250 U	250 U	11,600,000
MFAPES288FD	08/09/99	32	0-6	TOLTEST	250 U	250 U	120 J	200 J	61 J	250 U	250 U	12,800,000
MFAPES289	08/09/99	32	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES290	08/10/99	30	0-6	TOLTEST	250 U	250 U	250 U	250 U	250 U	250 U	250 U	10,800,000
MFAPES290FD	08/10/99	30	0-6	TOLTEST	250 U	250 U	250 U	250 U	250 U	250 U	250 U	11,000,000
MFAPES291	08/10/99	30	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES292	08/11/99	130	0-6	TOLTEST	250 U	250 U	250 U	250 U	250 U	250 U	250 U	12,800,000
MFAPES293	08/11/99	130	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES294	08/11/99	39	0-6	TOLTEST	250 U	250 U	400	300	250 U	250 U	250 U	11,100,000
MFAPES295	08/11/99	39	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES296	08/27/99	60	0-7	TOLTEST	1,000 U	1,000 U	1000 U	170 J	1,000 U	1,000 U	1,000 U	16,900,000
MFAPES296FD	08/27/99	60	0-7	TOLTEST	1,000 U	1,000 U	1000 U	158 J	1,000 U	0	1,000 U	13,000,000

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES271	06/21/99	78	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES272	06/21/99	78	sidewall	TOLTEST	**7,000 N***	101,000 N*	40 NU	15,000 N	14,000 N*	40	400 N*U	150 U
MFAPES273	06/21/99	78	sidewall	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES274	06/21/99	82	0-6	TOLTEST	**7,000 N***	110,000 N*	40 NU	18,000 N	15,000 N*	30 B	410 N*U	150 U
MFAPES275	06/21/99	82	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES276	06/21/99	83	0-6	TOLTEST	**9,000 N***	174,000 N*	100 NB	14,000 N	17,000 N*	40	1,000 N*B	150 U
MFAPES277	06/21/99	83	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES278	06/21/99	84	0-6	TOLTEST	**8,000 N***	150,000 N*	40 NU	17,000 N	16,000 N*	40 B	440 N*U	150 U
MFAPES279	06/21/99	84	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES280	06/21/99	92	0-6	TOLTEST	**10,000 N***	54,000 N*	40 NU	21,000 N	13,000 N*	80	390 N*U	140 U
MFAPES281	06/21/99	92	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES282	08/04/99	28	0-6	TOLTEST	**8,000**	76,000	40 U	16,000	19,000	60 N	390 U	140 U
MFAPES282FD	08/04/99	28	0-6	TOLTEST	**8,000**	70,000	30 U	22,000	14,000	40 N	370 U	130 U
MFAPES283	08/04/99	28	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES284	08/04/99	27	0-6	TOLTEST	**8,000**	76,000	170 B	17,000	16,000	80 N	360 U	130 U
MFAPES285	08/04/99	27	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES286	08/04/99	31	0-6	TOLTEST	**7,000**	72,000	30 U	14,000	15,000	60 N	370 U	130 U
MFAPES287	08/04/99	31	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES288	08/09/99	32	0-6	TOLTEST	*11,500 U*	68,500	580 U	16,300	11,600	24 J	23,000 U	1,200 U
MFAPES288FD	08/09/99	32	0-6	TOLTEST	**7,900 J**	52,000	580 U	19,600	11,800	14 J	23,000 U	1,200 U
MFAPES289	08/09/99	32	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES290	08/10/99	30	0-6	TOLTEST	**6,900 J**	238,000	570 U	24,300	14,000	110 U	22,700 U	1,100 U
MFAPES290FD	08/10/99	30	0-6	TOLTEST	*11,300 U*	321,000	560 U	18,700	12,500	110 U	22,500 U	1,100 U
MFAPES291	08/10/99	30	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES292	08/11/99	130	0-6	TOLTEST	**7,400 J**	54,700	540 U	38,100	34,600	110 U	21,400 U	1,100 U
MFAPES293	08/11/99	130	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES294	08/11/99	39	0-6	TOLTEST	**7,000 J**	110,000	620 U	17,900	13,400	20 J	24,600 U	1,200 U
MFAPES295	08/11/99	39	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES296	08/27/99	60	0-7	TOLTEST	**9,020 J**	73,500	634 U	19,600	18,100	58 J	25,400 U	1,270 U
MFAPES296FD	08/27/99	60	0-7	TOLTEST	**6,110 J**	75,500	666 U	15,100	18,100	44 J	26,600 U	1,330 U

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					VOLATILES						EXPLOSIVES
ANALYTE					MEK	MIBK	ACETONE	METHYLENE CHLORIDE	TOLUENE	XYLENES	DNB
IM Industrial Cleanup Goal (Human Health)					27,000,000	2,800,000	8,800,000	18,000	880,000	320,000	68,000
IM Residential Cleanup Goal (Human Health)					7,100,000	770,000	2,000,000	7,800	790,000	320,000	6,500
Units of Measurement					UG/KG	.UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY	**4,000,000						
MFAPES297	08/27/99	60	6	TOLTEST	14 U	7 U	42 B	7 U	7 U	7 U	N/A
MFAPES298	08/27/99	37	0-8	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	1,000 U
MFAPES299	08/27/99	37	6	TOLTEST	12 U	6 U	24 U	6 U	6 U	6 U	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES						
ANALYTE					HMX	NITROBENZENE	PETN	RDX	TNB	TETRYL	TNT
IM Industrial Cleanup Goal (Human Health)					34,000,000	94,000	250	17,000	34,000	6,800,000	64,000
IM Residential Cleanup Goal (Human Health)					3,300,000	18,000	250	4,000	3,300	650,000	15,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**40,000					
MFAPES297	08/27/99	60	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES298	08/27/99	37	0-8	TOLTEST	44,000 D	1,000 U	*2,500 U*	**272,000 D**	1,000 U	1,000 U	1,000 U
MFAPES299	08/27/99	37	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A

MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					EXPLOSIVES							METALS
ANALYTE					2,4-DNT	2,6-DNT	2-Am DNT	4-Am DNT	2-NT	3-NT	4-NT	ALUMINUM
IM Industrial Cleanup Goal (Human Health)					1,400,000	680,000	1,400,000	680,000	6,800,000	6,800,000	6,800,000	100,000,000
IM Residential Cleanup Goal (Human Health)					130,000	65,000	130,000	65,000	650,000	650,000	650,000	77,000,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY								
MFAPES297	08/27/99	60	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES298	08/27/99	37	0-8	TOLTEST	1,000 U	1,000 U	124 J	1,000 U	1,000 U	1,000 U	1,000 U	11,700,000
MFAPES299	08/27/99	37	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

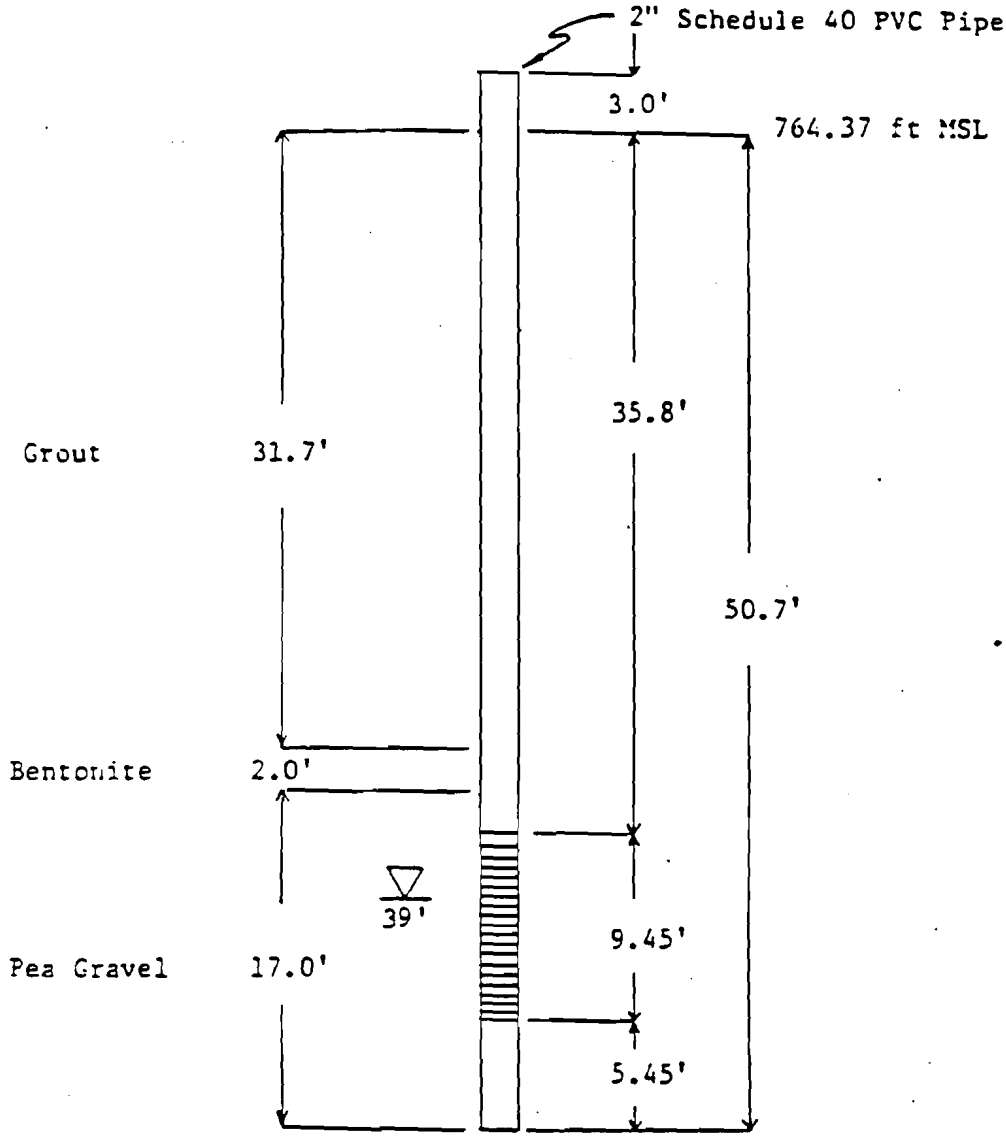
MINE FILL A POST-EXCAVATION SAMPLING RESULTS
NSWC CRANE

					METALS							
ANALYTE					ARSENIC	BARIUM	CADMIUM	CHROMIUM	LEAD	MERCURY	SELENIUM	SILVER
IM Industrial Cleanup Goal (Human Health)					2,400	100,000,000	850,000	450,000	1,000,000	510,000	8,500,000	8,500,000
IM Residential Cleanup Goal (Human Health)					320	5,300,000	38,000	210,000	400,000	23,000	380,000	380,000
Units of Measurement					UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG	UG/KG
SITE ID	SAMPLE DATE	GRID NUMBER	DEPTH (inches)	SAMPLED BY		**2,000,000	**20,000	**100,000	**100,000	**4,000		
MFAPES297	08/27/99	60	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A
MFAPES298	08/27/99	37	0-8	TOLTEST	*12,300 U*	59,400	615 U	17,100	13,800	9 J	24,600 U	1,230 U
MFAPES299	08/27/99	37	6	TOLTEST	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A

SWMU 16

BUILDING 146

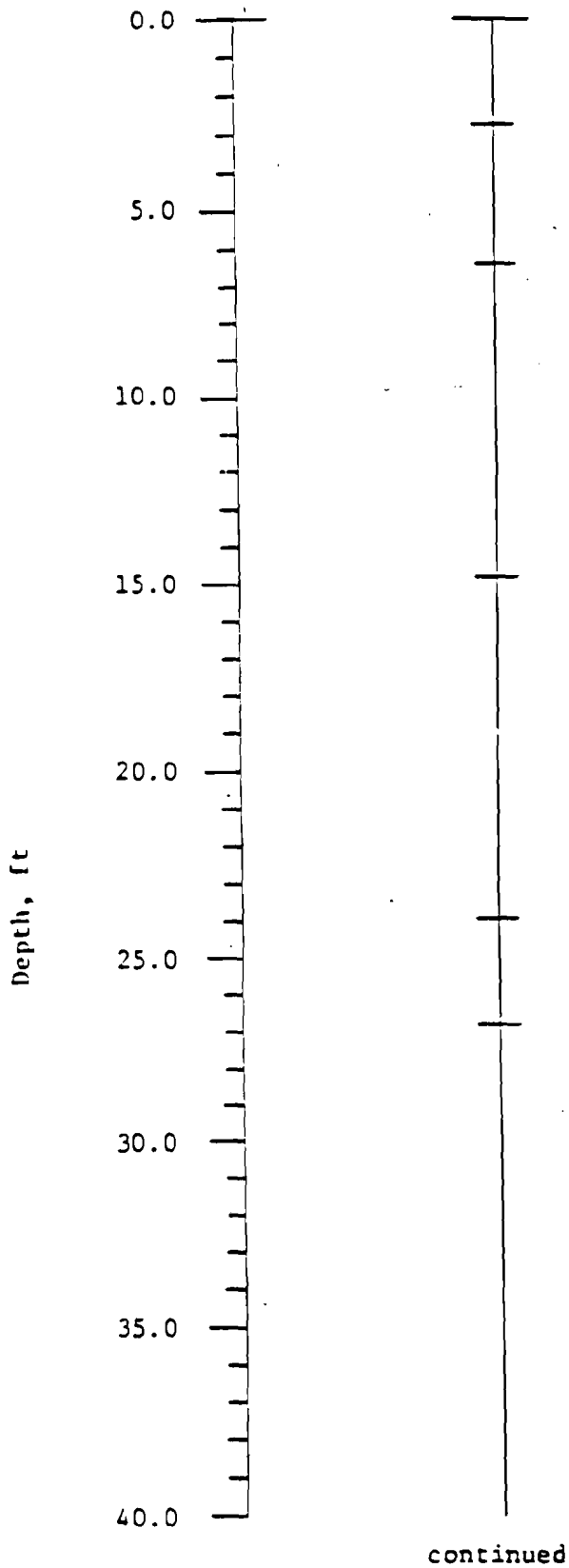
SOIL BORING DATA



Water Depth at Time of Drilling

Well Screen

NWSC, Crane, Indiana
Building 146-Ordnance
Demil Facility
Well Completion
Boring Number: WES-14-1-83



764.37 ft MSL

Sandy clay(CL): brown, soft, damp, very fine-grained sand, gravelly (sandstone).

Siltstone: brown to light grey, uniform, composition (quartz), soft to stiff, highly weathered damp.

Clay Shale: red, brown, black, orange brown, very thinly bedded (1/16"-1/4") highly weathered, soft to very stiff, brittle, and contains iron concreations or lenses.

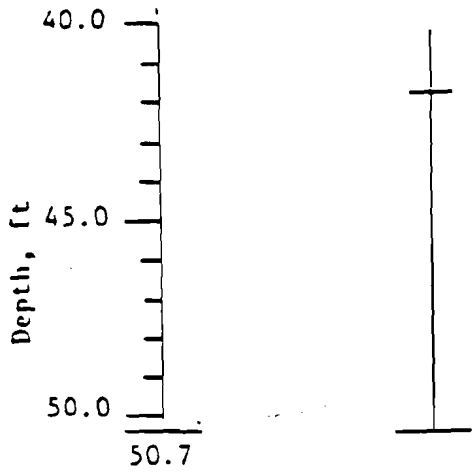
Shale: dark grey to black, weathered and oxidized in regions, very thinly bedded, contains micro subhedral grains of calcite.

Coal: black, soft to moderately hard, earth bituminous, brittle, blocky, contains limoni and approximately 5% sulfides (pyrite and chalcopyrite).

Sandstone: white to light grey, red, and brown, thinly bedded, fractured, oxidized, uniform, predominantly very fine-grained quartz sand, moderately to slightly friable, bedding dips approximately 15 degrees from horizontal with cross-bedding visible.

continued

NWSC, Crane, Indiana
Building 146-Ordnance Demil Facility
Lithology
Boring Number: WES-14-1-83

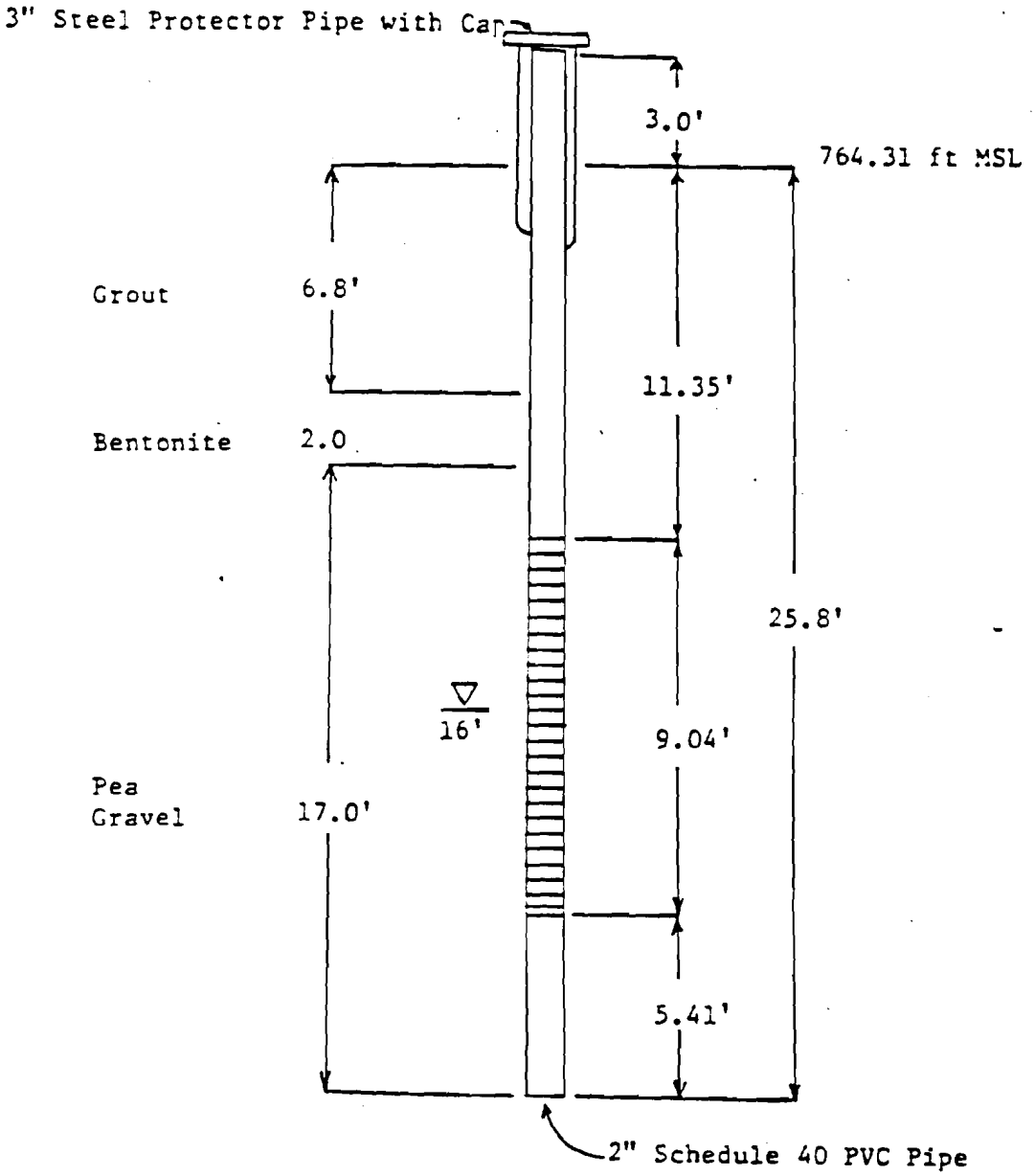


724.37 ft MSL

Sandstone (60%) and shale (40%): very fine sandstone interbedded with black soft shale ding consists of very fine lamina, randomly displaying irregular near shore deposition.

Shale: dark grey, sandy, uniform, soft

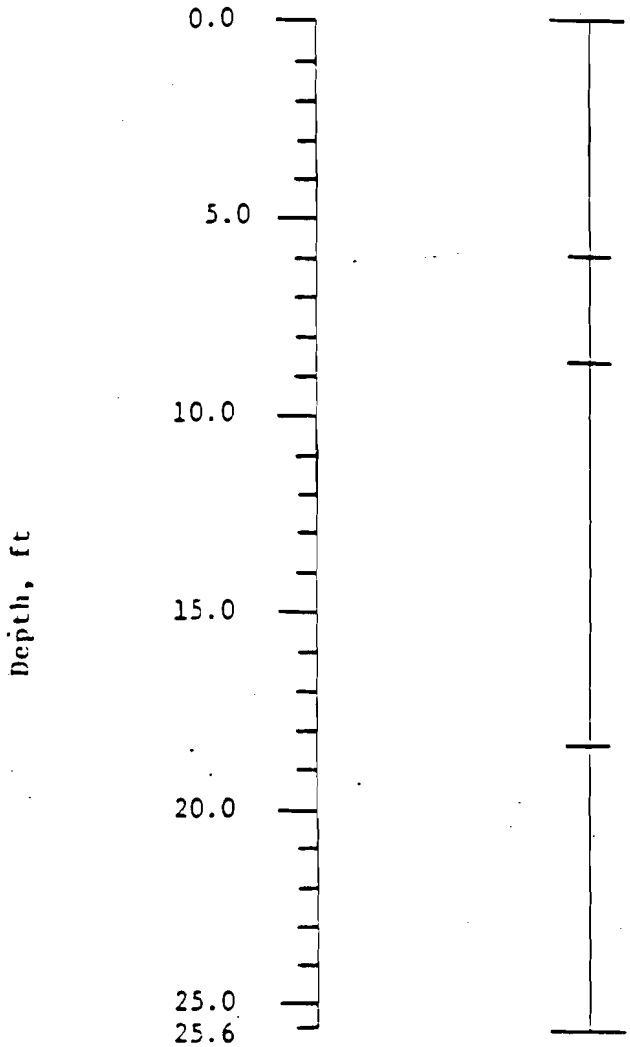
NWSC, Crane, Indiana
Building 146 Ordnance Demil Facil
Lithology
Boring Number: WES-14-1-83



▽ Water Depth at Time of Drilling

Well Screen

NWSC, Crane, Indiana
Building 146-Ordnance
Demil Facility
Well Completion
Boring Number: WES-14-2-83



764.31 ft MSL

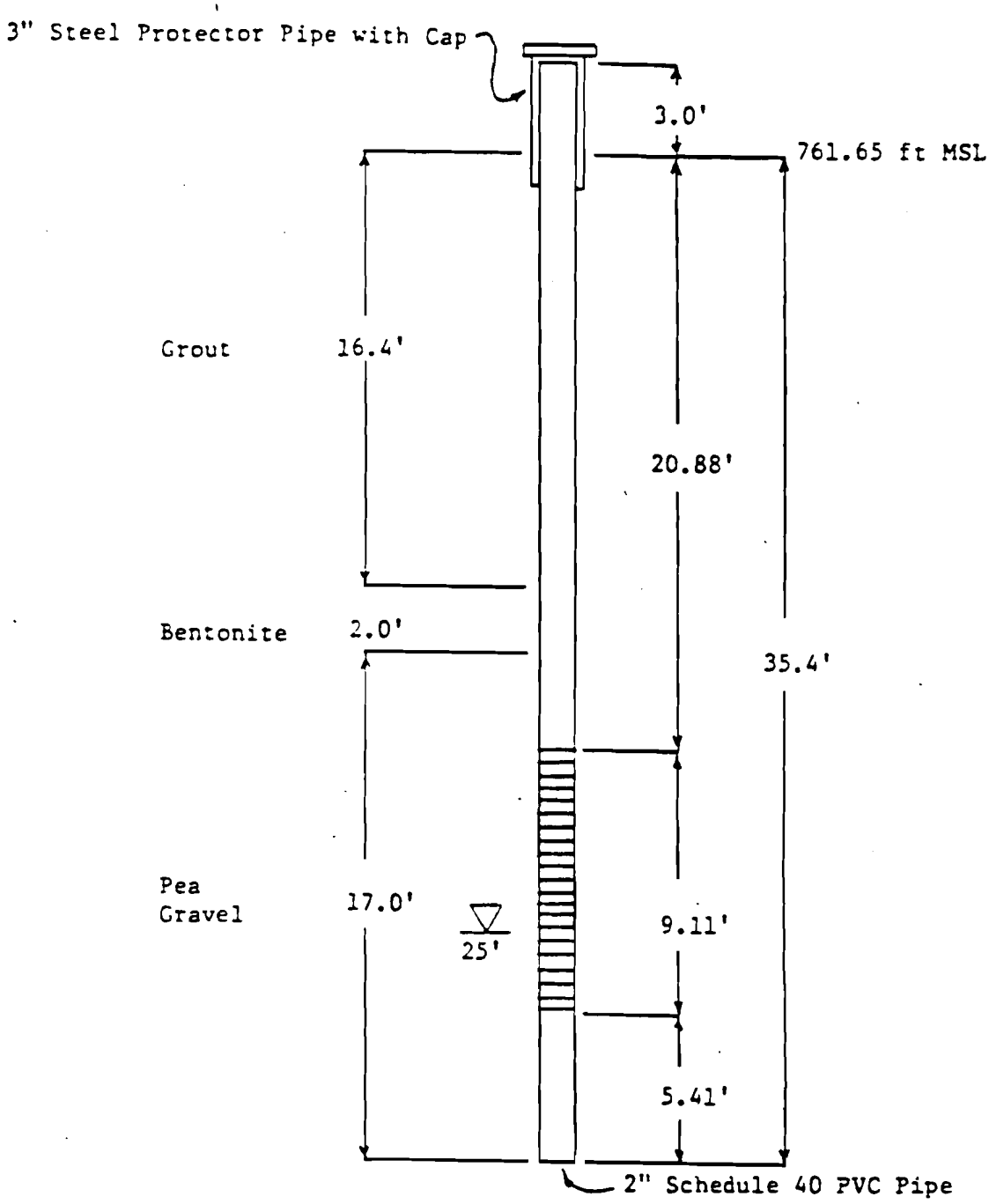
Clay(CL): brown, soft, damp, organic matter

Clayey sand(SC): brown, soft to stiff, very fine-grained sand, uniform, slightly damp.

Sandstone: brown, dark brown, very fine-grained sand (quartz), wet from 15 to 16 ft.

Shale: dark grey and brown, soft.

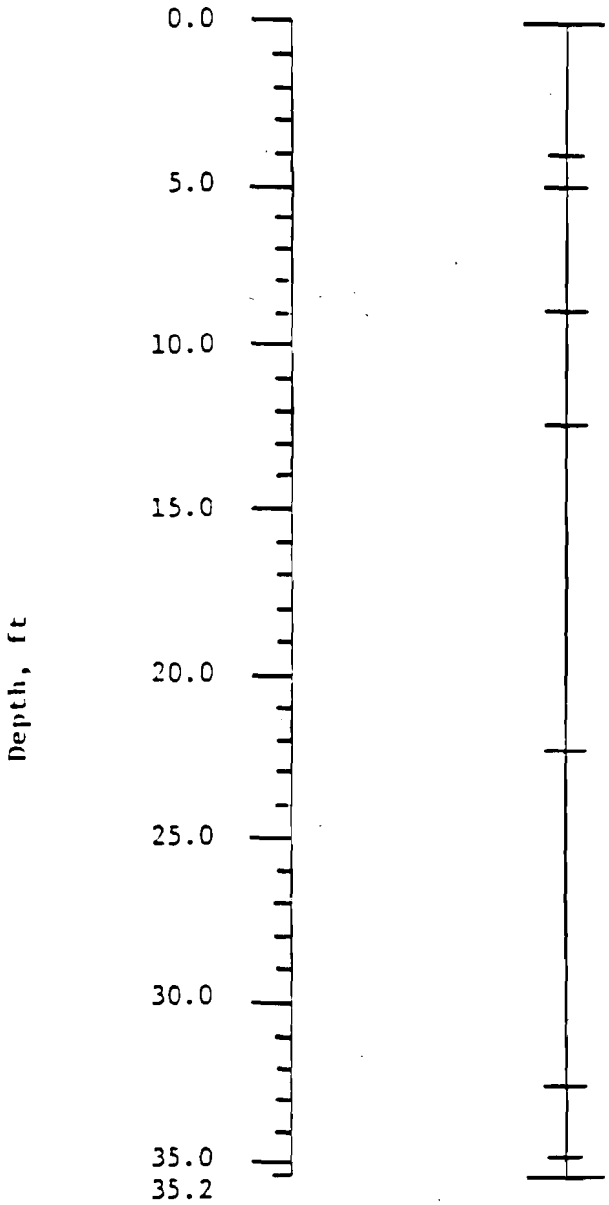
NWSC, Crane, Indiana
Building 146-Ordnance Demil Facility
Lithology
Boring Number: WES-14-2-83



Water Depth at Time of Drilling

Well Screen

NWSC, Crane, Indiana
Building 146-Ordnance
Demil Facility
Well Completion
Boring Number: WES-14-3-83



761.65 ft MSL

Clay (CL): brown, soft, sandy, damp, organic matter (roots)

Clay (CL): greenish grey, very soft, c

Sandstone: brown, soft, very fine-grair clay lenses

Shale: brown, weathered, hard from 8.8 11 ft (iron)

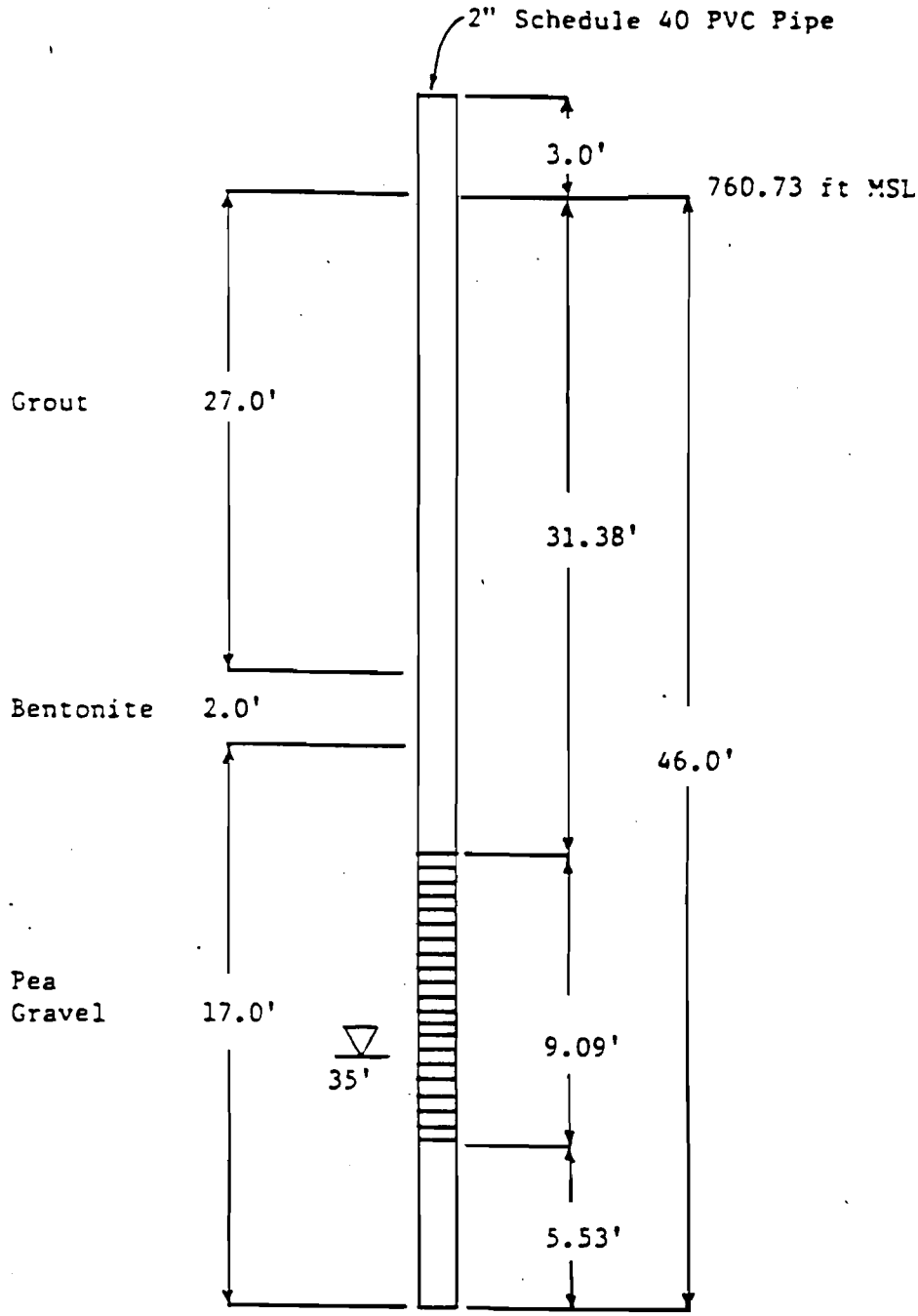
Sandstone: brown, grey (beginning at 15 ft), very fine-grained, soft

Shale: grey, dark grey, soft, water at 25 ft

Coal: black, soft (bituminous)

Shale: see above

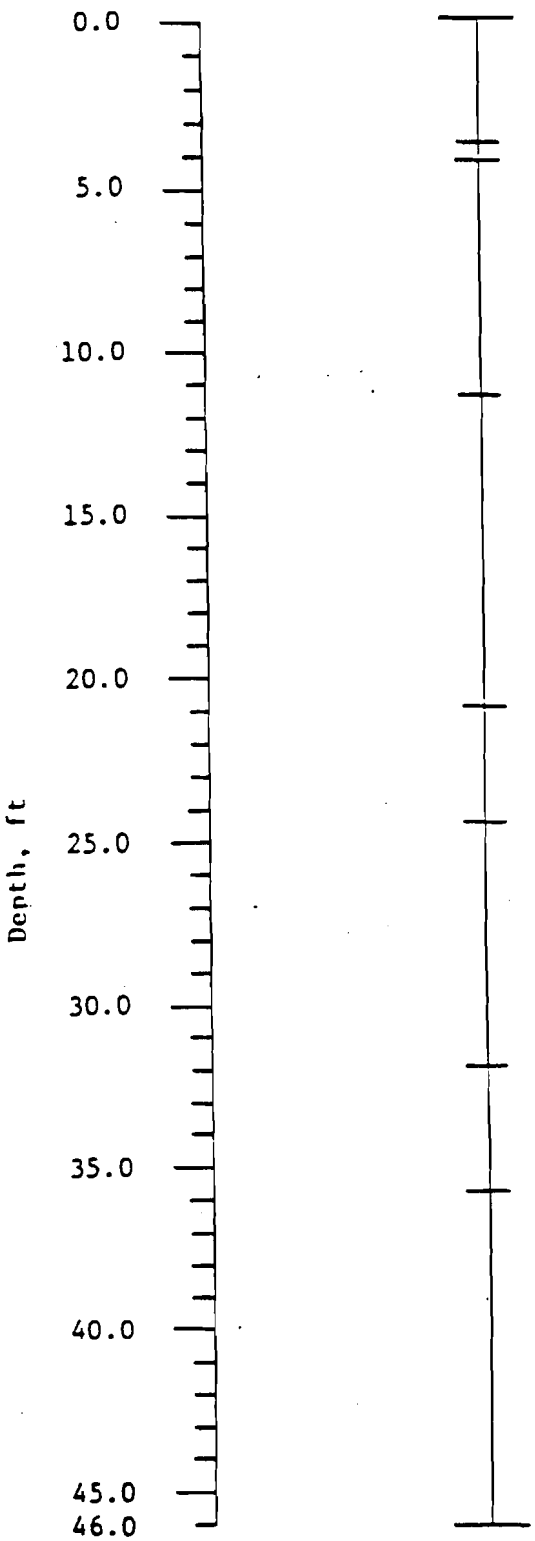
NWSC, Crane, Indiana
Building 146-Ordnance Demil Fa
Lithology
Boring Number: WES-14-3-03



Water Depth at Time of Drilling

Well Screen

NWSC, Crane, Indiana
Building 146-Ordnance
Demil Facility
Well Completion
Boring Number: WES-14-4-83



760.73 ft MSL

Sandy silt (ML): brown with grey streaks, soft, damp, very fine-grained sand.

Coal: black, soft, earthy

Siltstone: light to dark grey, light brown, soft.

Shale: grey, red (13.3 - 13.8 ft), soft

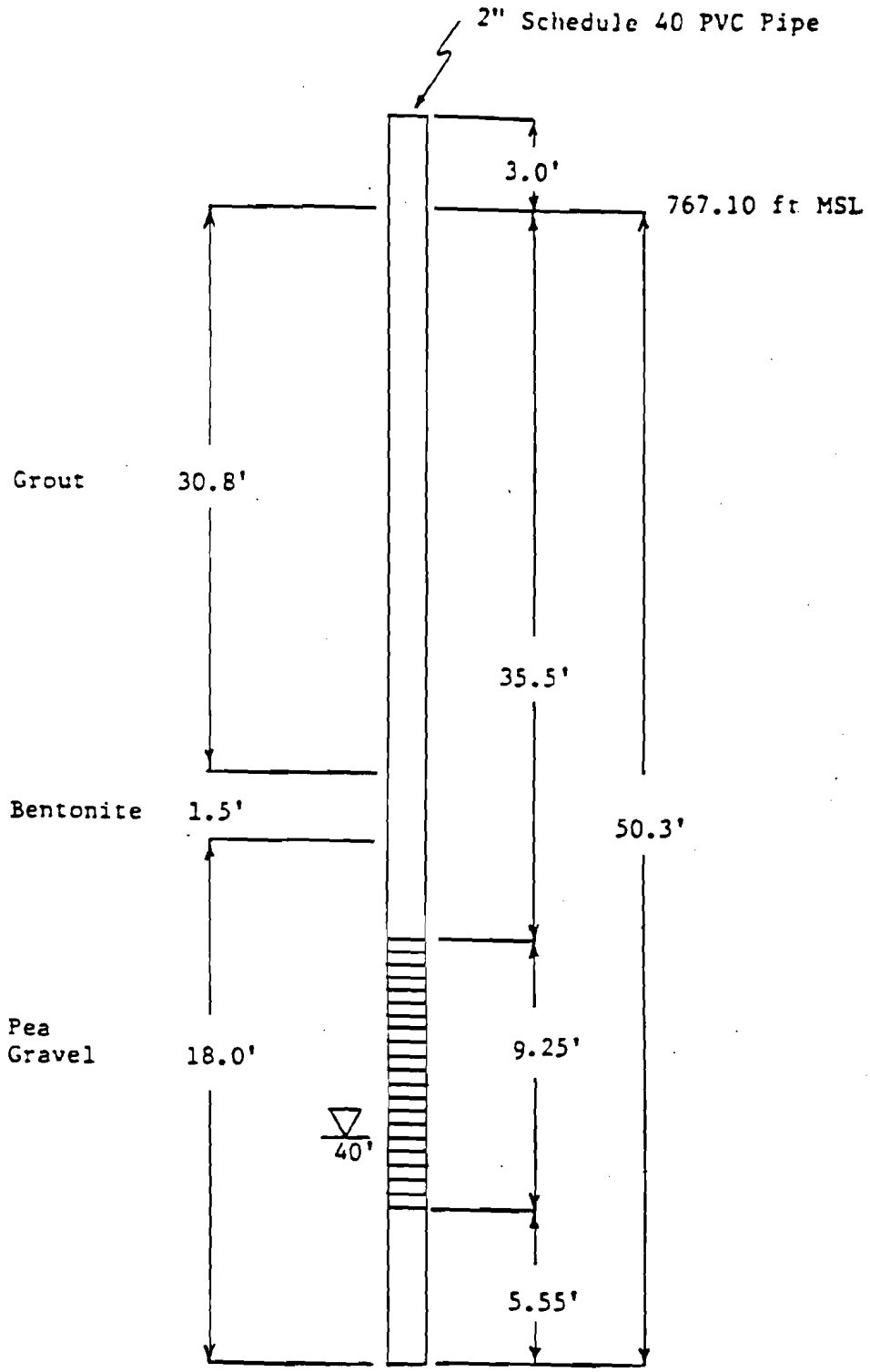
Sandstone and shale: grey to dark grey, soft thinly bedded, soft.

Shale: dark grey, soft

Coal: black, soft, wet between 35-36 ft (bituminous)

Sandstone and/or shale (?)- no cuttings, very wet

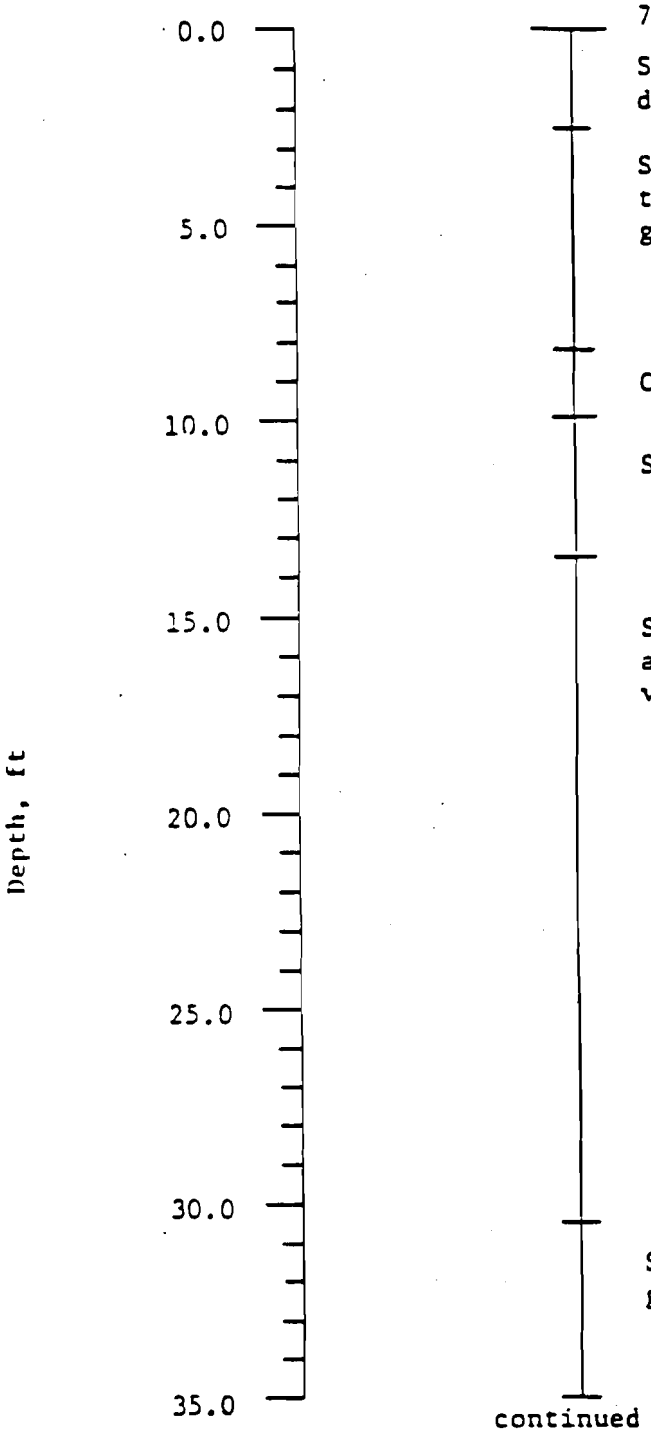
NWSC, Crane, Indiana
Building 146-Ordnance Demil Facility
Lithology
Boring Number: WES-14-4-83



Water Depth at Time of Drilling

Well Screen

NWSC, Crane, Indiana
Building 146-Ordnance
Demil Facility
Well Completion
Boring Number: WES-14-5-83



767.10 ft MSL

Sandy clay(CL): brown, soft to very stiff dry, sandy- very fine-grained sand.

Sandstone and clay: brown, reddish brown, thinly bedded, weathered, soft, very fine grained sandstone.

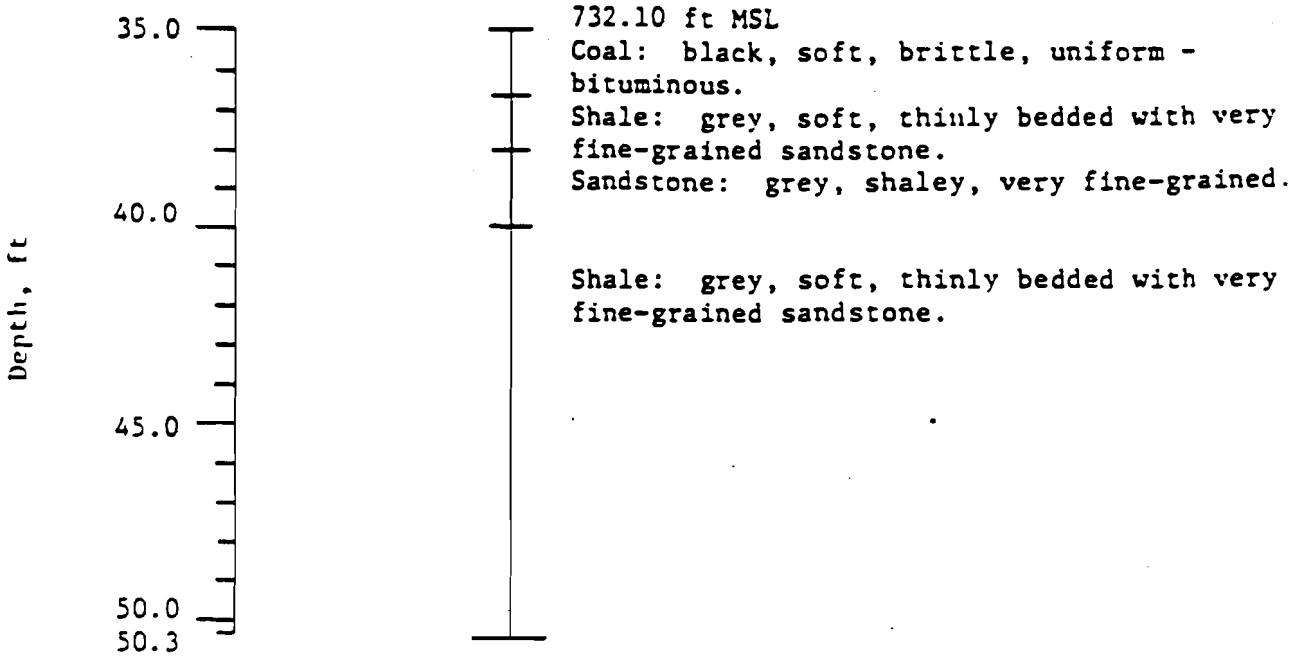
Coal: black, soft

Shale: light grey, soft, uniform

Sandstone and shale: light brown to brown and light grey to dark grey thinly bedded very fine-grained sandstone, soft.

Shale: grey, soft, uniform, sandy - fine-grained.

NWSC, Crane, Indiana
Building 146-Ordnance Demil Facility
Lithology
Boring Number: WES-14-5-83



NWSC, Crane, Indiana
Building 146-Ordnance Demil
Facility
Lithology
Boring Number: WES-14-5-83

APPENDIX B

TABULAR PRESENTATION OF RBSLs

APPENDIX B
TABLE 1
TABULAR PRESENTATION OF AQUEOUS RBSLs FOR RBTL SELECTION
NSWC, CRANE, INDIANA

Parameter	CAS	REGION 5 ECO DQLs SURFACE WATER (ug/L)	Footnote	FEDERAL MCLs (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER ACUTE (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER CHRONIC (ug/L)	Footnote	IDEM DEFAULT RESIDENTIAL GW CLOSURE LEVELS (ug/L)	Footnote	IDEM DEFAULT INDUSTRIAL GW CLOSURE LEVELS (ug/L)	Footnote	REGION 9 TAP WATER PRGS (ug/L)	Footnote	MINIMUM CRITERIA VALUE (ug/L)
Method 8330-EXPLOSIVES																
1,3,5-TRINITROBENZENE	99-35-4													1100	N	1100
1,3-DINITROBENZENE	99-65-0	2.36												3.6	N	2.36
2,4,6-TRINITROTOLUENE	118-96-7													2.2	C	2.2
2,4-DINITROTOLUENE	121-14-2	230								1.3(1)	C	4.2(1)	C	73	N	1.3
2,6-DINITROTOLUENE	606-20-2	42								1.3(1)	C	4.2(1)	C	36	N	1.3
2-AMINO-4,6-DINITROTOLUENE	35572-78-2													2.2(2)	N	2.2
2-NITROTOLUENE	88-72-2													61	N	61
4-NITROTOLUENE	99-99-0													61	N	61
HMX	2691-41-0													1800	N	1800
NITROBENZENE	98-95-3	740								4.3	N	51	N	3.4	N	3.4
RDX	121-82-4													0.61	C	0.61
TETRYL	479-45-8													360	N	360
Method LAUCKS HPLC-EXPLOSIVES																
PICRAMIC ACID	96-91-3															
PICRIC ACID	88-89-1															
METALS																
Method 6020 ICP/MS-METALS																
ANTIMONY	7440-36-0	31		6						6	N	41	N	15	N	6
ARSENIC	7440-38-2	53		5		340		150		50	C	50	C	0.045	C	0.045
BARIUM	7440-39-3	5000		2000						2000	N	7200	N	2600	N	2000
BERYLLIUM	7440-41-7	7.6		4						4	N	4	N	73	N	4
CADMIUM	7440-43-9	0.66		5		4.3	3	2.2	3	5	N	51	N	18	N	0.66
CHROMIUM (TOTAL)	7440-47-3	42		100		16	4	11	4	100	N	510	N	110(4)	N	11
COBALT	7440-48-4	5												2200	N	5
COPPER	7440-50-8	5		1300	5,6	13	3	9	3	1300	N	3800	N	1400	N	5
LEAD	7439-92-1	1.3		15	5	65	3	2.5	3	15		42				1.3
LITHIUM	743993-2													730	N	730
MERCURY (SW-846 METHOD 7470A/7471A)	7439-97-6	0.0013		2		1.4		0.77		2	N	31	N	11	N	0.0013
NICKEL	7440-02-0	29				470	3	52	3	100	N	2000	N	730	N	29
SELENIUM	7782-49-2	5		50				5.0		50	N	510	N	180	N	5
SILVER	7440-22-4	1		100	7	3.4	3			180	N	510	N	180	N	1
STRONTIUM	7440-24-6													22000	N	22000
THALLIUM	7440-28-0	0.56		2						2	N	9	N	2.4	N	0.56
TIN	7440-31-5	73												22000	N	73
VANADIUM	7440-62-2	19												260	N	19
ZINC	7440-66-6	58.9		5000	7	120	3	120	3	11000	N	31000	N	11000	N	58.9
ALUMINUM	7429-90-5			50-200	7	750		87						36000	N	50
IRON	7439-89-6			300	7									11000	N	300
MAGNESIUM	7439-95-4															
MANGANESE	7439-96-5			50	7									880	N	50
CALCIUM	7440-70-2															
POTASSIUM	7440-09-7															
SODIUM	7440-23-5															
TITANIUM	7440-32-6															
VOLATILES																
Method 8260B-VOC																

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NSWC, CRANE, INDIANA

Parameter	CAS	REGION 5 ECO DQLs SURFACE WATER (ug/L)	Footnote	FEDERAL MCLs (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER ACUTE (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER CHRONIC (ug/L)	Footnote	IDEM DEFAULT RESIDENTIAL GW CLOSURE LEVELS (ug/L)	Footnote	IDEM DEFAULT INDUSTRIAL GW CLOSURE LEVELS (ug/L)	Footnote	REGION 9 TAP WATER PRGS (ug/L)	Footnote	MINIMUM CRITERIA VALUE (ug/L)
1,1,1,2-TETRACHLOROETHANE	630-20-6	90.25								6.9	C	110	C	0.43	C	0.43
1,1,1-TRICHLOROETHANE	71-55-6	88		200						200	N	9200	N	540	N	88
1,1,2,2-TETRACHLOROETHANE	79-34-5	13								0.9	C	14	C	0.055	C	0.055
1,1,2-TRICHLOROETHANE	79-00-5	650		5						5	C	50	C	0.2	C	0.2
1,1-DICHLOROETHANE	75-34-3	47								990	N	10000	N	810	N	47
1,1-DICHLOROETHENE	75-35-4	78		7						7	C	7	C	0.046	C	0.046
1,2,3-TRICHLOROPROPANE	96-18-4	12.11												0.0016	C	0.0016
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8	11.2		0.2										0.048	C	0.048
1,2-DIBROMOETHANE	106-93-4	22.5		0.05										0.00076	C	0.00076
1,2-DICHLOROETHANE	107-06-2	190		5						5	C	31	C	0.12	C	0.12
1,2-DICHLOROPROPANE	78-87-5	380		5						5	C	42	C	0.16	C	0.16
2-BUTANONE	78-93-3	7100								2500	N	61000	N	1900	N	1900
2-HEXANONE	591-78-6	1710												1500(2)	N	1500
3-CHLOROPROPENE	107-05-1													1800	N	1800
4-METHYL-2-PENTANONE	108-10-1	3680												160	N	160
ACETONE	67-64-1	78000								770	N	10000	N	610	N	610
ACROLEIN	107-02-8	0.205								0.056	N	2000	N	0.042	N	0.042
ACRYLONITRILE	107-13-1													0.039	C	0.039
BENZENE	71-43-2	114		5						5	C	99	C	0.35	C	0.35
BROMODICHLOROMETHANE	75-27-4			80	8					100	C	100	C	0.18	C	0.18
BROMOFORM	75-25-2	466		80	8					100	C	360	C	8.5	C	8.5
BROMOMETHANE	74-83-9									11	N	140	N	8.7	N	8.7
CARBON DISULFIDE	75-15-0	84.1								1300	N	10000	N	1000	N	84.1
CARBON TETRACHLORIDE	56-23-5	5.9		5						5	C	22	C	0.17	C	0.17
CHLOROBENZENE	108-90-7	10		100						100	N	2000	N	110	N	10
CHLORODIBROMOMETHANE	124-48-1			80	8									0.13	C	0.13
CHLOROETHANE	75-00-3	230000												4.6	C	4.6
CHLOROFORM	67-66-3	79		80	8					100	C	470	C	0.16	C	0.16
CHLOROMETHANE	74-87-3													1.5	C	1.5
CHLOROPRENE	126-99-8													14	N	14
CIS-1,2-DICHLOROETHENE	156-59-2	78		70						70	N	1000	N	61	N	61
CIS-1,3-DICHLOROPROPENE	10061-01-5				9											
DIBROMOMETHANE	74-95-3													61	N	61
DICHLORODIFLUOROMETHANE	75-71-8													390	N	390
ETHYL METHACRYLATE	97-63-2													550	N	550
ETHYLBENZENE	100-41-4	17.2		700						700	N	10000	N	1300	N	17.2
METHACRYLONITRILE	126-98-7													1	N	1
METHYL IODIDE	74-88-4															
METHYL METHACRYLATE	80-62-6	2800												1400	N	1400
METHYLENE CHLORIDE	75-09-2	430		5						5	C	380	C	4.3	C	4.3
STYRENE	100-42-5	56		100						100	N	20000	N	1600	N	56
TETRACHLOROETHENE	127-18-4	8.9		5						5	C	55	C	1.1	C	1.1
TOLUENE	108-88-3	253		1000						1000	N	20000	N	720	N	253
TOTAL XYLENES	1330-20-7	117		10000						10000	N	180000	N	1400	N	117
TRANS-1,2-DICHLOROETHENE	156-60-5	310		100						100	N	2000	N	120	N	100
TRANS-1,3-DICHLOROPROPENE	10061-02-6	7.9			9									0.4	C	0.4
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6															

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TRICHLOROETHENE	79-01-6	75		5						5	C	260	C	1.6	C	1.6
TRICHLOROFLUOROMETHANE	75-69-4													1300	N	1300
VINYL ACETATE	108-05-4	248.03								550	N	100000	N	410	N	248.03
VINYL CHLORIDE	75-01-4	9.2		2						2	C	2	C	0.041	C	0.041
Method 8015B-VOC																
1,4-DIOXANE	123-91-1													6.1	C	6.1
ACETONITRILE	75-05-8	30000												79	N	79
ISOBUTANOL	78-83-1	34800												1800	N	1800
PROPIONITRILE	107-12-0	6080														6080
SEMIVOLATILES																
Method 8270C-SVOC																
1,2,4,5-TETRACHLOROBENZENE	95-94-3	26.24												11	N	11
1,2,4-TRICHLOROBENZENE	120-82-1	69.2		70						70	N	1000	N	190	N	69.2
1,2-DICHLOROBENZENE	95-50-1	11		600						600	N	9200	N	370	N	11
1,3-DICHLOROBENZENE	541-73-1	87								22	N	3100	N	5.5	N	5.5
1,4-DICHLOROBENZENE	106-46-7			75						75	C	120	C	0.5	C	0.5
1,4-NAPHTHOQUINONE	130-15-4	0.044														0.044
1,4-PHENYLENEDIAMINE	106-50-3													6900	N	6900
1-NAPHTHYLAMINE	134-32-7	0.67														0.67
2,3,4,6-TETRACHLOROPHENOL	58-90-2	14.06												1100	N	14.06
2,4,5-TRICHLOROPHENOL	95-95-4									3600	N	10000	N	3600	N	3600
2,4,6-TRICHLOROPHENOL	88-06-2	2								77	C	260	C	6.1	C	2
2,4-DICHLOROPHENOL	120-83-2	18								110	N	310	N	110	N	18
2,4-DIMETHYLPHENOL	105-67-9	100.17								730	N	2000	N	730	N	100.17
2,4-DINITROPHENOL	51-28-5	4.07								73	N	200	N	73	N	4.07
2,4-DINITROTOLUENE	121-14-2	230												73	N	73
2,6-DICHLOROPHENOL	87-65-0															
2-ACETYLAMINOFLUORENE	53-96-3	534.97														534.97
2-CHLORONAPHTHALENE	91-58-7	0.396												490	N	0.396
2-CHLOROPHENOL	95-57-8	8.8								38	N	510	N	30	N	8.8
2-METHYLPHENOL	95-48-7									1800	N	5100	N	1800	N	1800
2-NAPHTHYLAMINE	91-59-8															
2-NITROANILINE	88-74-4													2.1	N	2.1
2-NITROPHENOL	88-75-5	13.5												290(10)	N	13.5
2-PICOLINE	109-06-8	3790														3790
3,3'-DICHLOROBENZIDINE	91-94-1	99.75								1.9	C	6.4	C	0.15	C	0.15
3,3'-DIMETHYLBENZIDINE	119-93-7													0.0073	C	0.0073
3-METHYLCHOLANTHRENE	56-49-5	0.0891														0.0891
3-METHYLPHENOL	108-39-4													1800	N	1800
3-NITROANILINE	99-09-2													2.1	N	2.1
4,6-DINITRO-2-METHYLPHENOL	534-52-1	2.3														2.3
4-AMINOBIPHENYL	92-67-1															
4-BROMOPHENYL PHENYL ETHER	101-55-3	1.5														1.5
4-CHLORO-3-METHYLPHENOL	59-50-7	34.79														34.79
4-CHLOROANILINE	106-47-8	231.97								150	N	410	N	150	N	150
4-CHLOROPHENYL PHENYL ETHER	7005-72-3															
4-METHYLPHENOL	106-44-5													180	N	180

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4-NITROANILINE	100-01-6													2.1	N	2.1
4-NITROPHENOL	100-02-7	35												290	N	35
4-NITROQUINOLINE-1-OXIDE	56-57-5															
5-NITRO-O-TOLUIDINE	99-55-8													2	C	2
7,12-DIMETHYLBENZ(A)ANTHRACENE	57-97-6	0.548														0.548
A,A-DIMETHYLPHENETHYLAMINE	122-09-8													36	N	36
ACETOPHENONE	98-86-2	687.89												0.042	N	0.042
ANILINE	62-53-3	0.44												12	C	0.44
ARAMITE	140-57-8	3.09												2.7	C	2.7
BENZYL ALCOHOL	100-51-6	281.24												11000	N	281.24
BIS(2-CHLOROETHOXY)METHANE	111-91-1	6400														6400
BIS(2-CHLOROETHYL)ETHER	111-44-4	1140								0.15	C	2.6	C	0.0098	C	0.0098
BIS(2-CHLOROISOPROPYL)ETHER	39638-32-9															
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	2.1		6						6	C	200	C	4.8	C	2.1
BUTYL BENZYL PHTHALATE	85-68-7	49								2700	N	2700	N	7300	N	49
CHLOROBENZILATE	510-15-6	7.16												0.25	C	0.25
DIALLATE	2303-16-4	29												1.1	C	1.1
DIBENZOFURAN	132-64-9	20												24	N	20
DIETHYL PHTHALATE	84-66-2	3								29000	N	82000	N	29000	N	3
DIMETHYL PHTHALATE	131-11-3	73												360000	N	73
DI-N-BUTYL PHTHALATE	84-74-2	3								3600	N	10000	N	3600	N	3
DI-N-OCTYL PHTHALATE	117-84-0	30								20	N	20	N	730	N	20
DIPHENYLAMINE	122-39-4	412.51												910	N	412.51
ETHYL METHANE SULFONATE	62-50-0															
HEXACHLOROBENZENE	118-74-1	0.00024		1						1	C	1.8	C	0.042	C	0.00024
HEXACHLOROBUTADIENE	87-68-3	0.223								7.3	N	20	N	0.86	C	0.223
HEXACHLOROCYCLOPENTADIENE	77-47-4	77.04		50						50	N	720	N	260	N	50
HEXACHLOROETHANE	67-72-1	30.5								36	N	100	N	4.8	C	4.8
HEXACHLOROPROPENE	1888-71-7	20														20
ISODRIN	465-73-6	0.0309														0.0309
ISOPHORONE	78-59-1	900								900	C	3000	C	71	C	71
ISOSAFROLE	120-58-1															
KEPONE	143-50-0	0.132												0.0037	C	0.0037
METHAPYRILENE	91-80-5															
METHYL METHANE SULFONATE	66-27-3															
N-NITROSODIETHYLAMINE	55-18-5	767.94												0.00045	C	0.00045
N-NITROSODIMETHYLAMINE	62-75-9													0.0013	C	0.0013
N-NITROSODI-N-BUTYLAMINE	924-16-3	1000												0.002	C	0.002
N-NITROSO-DI-N-PROPYLAMINE	621-64-7									0.12	C	0.41	C	0.0096	C	0.0096
N-NITROSOMETHYLETHYLAMINE	10595-95-6													0.0031	C	0.0031
N-NITROSOMORPHOLINE	59-89-2															
N-NITROSOPIPERIDINE	100-75-4															
N-NITROSOPYRROLIDINE	930-55-2													0.032	C	0.032
O,O,O-TRIETHYL PHOSPHOROTHIOATE	126-68-1	58.25														58.25
O-TOLUIDINE	95-53-4													0.28	C	0.28
P-(DIMETHYLAMINO)AZOBENZENE	60-11-7															
PENTACHLOROBENZENE	608-93-5	0.47												29	N	0.47

APPENDIX B
TABLE 1
TABULAR PRESENTATION OF AQUEOUS RBSLs FOR RBTL SELECTION
NSWC, CRANE, INDIANA

Parameter	CAS	REGION 5 ECO DQLs SURFACE WATER (ug/L)	Footnote	FEDERAL MCLs (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER ACUTE (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER CHRONIC (ug/L)	Footnote	IDEM DEFAULT RESIDENTIAL GW CLOSURE LEVELS (ug/L)	Footnote	IDEM DEFAULT INDUSTRIAL GW CLOSURE LEVELS (ug/L)	Footnote	REGION 9 TAP WATER PRGS (ug/L)	Footnote	MINIMUM CRITERIA VALUE (ug/L)
PENTACHLOROETHANE	76-01-7	56.42														56.42
PENTACHLORONITROBENZENE	82-68-8	50												0.26	C	0.26
PHENACETIN	62-44-2	6.3														6.3
PHENOL	108-95-2	100								22000	N	61000	N	22000	N	100
PRONAMIDE	23950-58-5	160												2700	N	160
PYRIDINE	110-86-1	2380												36	N	36
SAFROLE	94-59-7	40														40
Method SELECTIVE ION MONITORING-SVOC																
2-METHYLNAPHTHALENE	91-57-6	329.55								8.3	11	2000	11	6.2	11	6.2
ACENAPHTHENE	83-32-9	9.9								460	N	4200	N	370	N	9.9
ACENAPHTHYLENE	208-96-8	4840								460	12	4200	12	370	12	370
ANTHRACENE	120-12-7	0.029								43	N	43	N	1800	N	0.029
BENZO(A)ANTHRACENE	56-55-3	0.839								1.2	C	3.9	C	0.092	C	0.092
BENZO(A)PYRENE	50-32-8	0.014		0.2						0.2	C	0.39	C	0.0092	C	0.0092
BENZO(B)FLUORANTHENE	205-99-2	9.07								1.2	C	1.5	C	0.092	C	0.092
BENZO(G,H,I)PERYLENE	191-24-2	7.64								140	13	140	13	180	13	7.64
BENZO(K)FLUORANTHENE	207-08-9	0.0056								0.8	C	0.8	C	0.92	C	0.0056
CHRYSENE	218-01-9	0.033								1.6	C	1.6	C	9.2	C	0.033
DIBENZO(A,H)ANTHRACENE	53-70-3	0.0016								0.12	C	0.39	C	0.0092	C	0.0016
FLUORANTHENE	206-44-0	8.1								210	N	210	N	1500	N	8.1
FLUORENE	86-73-7	3.9								310	N	2000	N	240	N	3.9
INDENO(1,2,3-CD)PYRENE	193-39-5	4.31								0.022	C	0.022	C	0.092	C	0.022
NAPHTHALENE	91-20-3	44								8.3	N	2000	N	6.2	N	6.2
PHENANTHRENE	85-01-8	2.1								140	13	140	13	180	13	2.1
PYRENE	129-00-0	0.3								140	N	140	N	180	N	0.3
Method 8082-ORGANOCHLORINE PCBs																
AROCLOR-1016	12674-11-2	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.96	C	0.000029
AROCLOR-1221	11104-28-2	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.034	C	0.000029
AROCLOR-1232	11141-16-5	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.034	C	0.000029
AROCLOR-1242	53469-21-9	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.034	C	0.000029
AROCLOR-1248	12672-29-6	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.034	C	0.000029
AROCLOR-1254	11097-69-1	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.034	C	0.000029
AROCLOR-1260	11096-82-5	0.000029	14	0.5	14			0.014	14	0.5(14)	C	1.4(14)	C	0.034	C	0.000029
Method 8151A-HERBICIDES																
HEXACHLOROPHENE	70-30-4	0.228												11	N	0.228
PENTACHLOROPHENOL	87-86-5	5.23		1		19		15		1	C	24	C	0.56	C	0.56
DIOXINS/FURANS																
Method 8290-DIOXINS/FURANS																
1,2,3,4,6,7,8,9-OCDD	3268-87-9	2.78E-07												0.0045	C	0.000000278
1,2,3,4,6,7,8,9-OCDF	39001-02-0	0.00129												0.0045	C	0.00129
1,2,3,4,6,7,8-HPCDD	35822-46-9	2.78E-07												0.000045	C	0.000000278
1,2,3,4,6,7,8-HPCDF	67562-39-4	0.00129												0.000045	C	0.000045
1,2,3,4,7,8,9-HPCDF	55673-89-7	0.00129												0.000045	C	0.000045
1,2,3,4,7,8-HXCDD	39227-28-6	2.78E-07												0.0000045	C	0.000000278
1,2,3,4,7,8-HXCDF	70648-26-9	0.00129												0.0000045	C	0.0000045
1,2,3,6,7,8-HXCDD	57653-85-7	2.78E-07												0.0000045	C	0.000000278
1,2,3,6,7,8-HXCDF	57117-44-9	0.00129												0.0000045	C	0.0000045

APPENDIX B
TABLE 1
TABULAR PRESENTATION OF AQUEOUS RBSLs FOR RBTL SELECTION
NSWC, CRANE, INDIANA

Parameter	CAS	REGION 5 ECO DQLs SURFACE WATER (ug/L)	Footnote	FEDERAL MCLs (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER ACUTE (ug/L)	Footnote	FEDERAL AWQCs FRESHWATER CHRONIC (ug/L)	Footnote	IDEM DEFAULT RESIDENTIAL GW CLOSURE LEVELS (ug/L)	Footnote	IDEM DEFAULT INDUSTRIAL GW CLOSURE LEVELS (ug/L)	Footnote	REGION 9 TAP WATER PRGS (ug/L)	Footnote	MINIMUM CRITERIA VALUE (ug/L)
1,2,3,7,8,9-HXCDD	19408-74-3	2.78E-07												0.0000045	C	0.000000278
1,2,3,7,8,9-HXCDF	72918-21-9	0.00129												0.0000045	C	0.0000045
1,2,3,7,8-PECDD	40321-76-4	2.78E-07												0.00000045	C	0.000000278
1,2,3,7,8-PECDF	57117-41-6	0.00129												0.000009	C	0.000009
2,3,4,6,7,8-HXCDF	60851-34-5	0.00129												0.0000045	C	0.0000045
2,3,4,7,8-PECDF	57117-31-4	0.00129												0.0000009	C	0.0000009
2,3,7,8-TCDD	1746-01-6	0.0000003		0.00003										0.00000045	C	0.0000003
2,3,7,8-TCDF	51207-31-9	0.00129												0.0000045	C	0.0000045
TOTAL HPCDD	37871-00-4															
TOTAL HPCDF	38998-75-3															
TOTAL HXCDD	34465-46-8													0.0000011		0.0000011
TOTAL HXCDF	55684-94-1															
TOTAL PECDD	36088-22-9															
TOTAL PECDF	30402-15-4															
TOTAL TCDD	41903-57-5															
TOTAL TCDF	55722-27-5															
MISCELLANEOUS PARAMETERS																
TOTAL ORGANIC CARBON - SW-846 METHOD 9060	TTNUS003															
PERCHLORATE - METHOD 314	14797-73-0															
NITRATE + NITRITE AS NITRATE EPA 353.2	14797-55-8			10000										10000	N	10000
HARDNESS - METHOD 23040B	TTNUS022															
PH - METHOD 9045C	TTNUS002			6.5 - 8.5	7			6.5-9.0								
TOTAL SUSPENDED SOLIDS - METHOD 160.2	TTNUS044															
NITRITE - METHOD 9081	14797-65-0			1000										1000	N	1000
CYANIDE - METHOD 9012A	57-12-5	5.2		200		22		5.2						6.2	N	5.2
AMMONIA EPA 350.1	7664-41-7													210(2)	N	

Footnotes:
1 Dinitrotoulene Mixture.
2 EPA Region 3 RBC presented.
3 Criteria are hardness dependent.
4 Hexavalent Chromium.
5 Action Level.
6 Secondary MCL for copper is 1,000 ug/L.
7 Secondary MCL based on aesthetic drinking water qualities (i.e., color, odor, taste, etc.).
8 1994 Proposed rule for Disinfectants and Disinfection By-Products. Total for all trihalomethanes combined cannot exceed 80 ug/L.
9 1,3-Dichloropropene.
10 Value for 4-nitrophenol presented.
11 Value for naphthalene presented.
12 Value for acenaphthene presented.
13 Value for pyrene presented.
14 Total for all Aroclor congeners.

APPENDIX B
Table 2
Tabular Presentation of Soil and Sediment RBSLs for RBTL Selection
NSWC Crane, Crane, Indiana

PARAMETER	CAS	EPA GENERIC SSLs INGESTION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs INHALATION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs MIGRATION TO GW (mg/kg) DAF=1	FOOTNOTE	REGION 5 ECO DQLs SOIL (mg/kg)	FOOTNOTE	REGION 5 ECO DQLs SEDIMENT (mg/kg)	FOOTNOTE	IDEM DEFAULT RESIDENTIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	IDEM DEFAULT INDUSTRIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR INDUSTRIAL LAND USE (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR RESIDENTIAL LAND USE (mg/kg)	FOOTNOTE	MINIMUM CRITERIA VALUE SOIL (mg/kg)	MINIMUM CRITERIA VALUE SEDIMENT (mg/kg)	MINIMUM CRITERIA VALUE SOIL AND SEDIMENT (mg/kg)
EXPLOSIVES																						
SW-846 METHOD 8330																						
1,3,5-TRINITROBENZENE	99-35-4							---		---						26000	N	1800	N	1800	1800	1800
1,3-DINITROBENZENE	99-65-0							0.6547		0.000924						88	N	6.1	N	0.6547	0.000924	0.000924
2,4,6-TRINITROTOLUENE	118-96-7							---		---						82	C	16	C	16	16	16
2,4-DINITROTOLUENE	121-14-2	0.9				0.00004		1.28		0.07513		0.0085(1)	C	0.028(1)	C	1800	N	120	N	0.00004	0.00004	0.00004
2,6-DINITROTOLUENE	606-20-2	0.9				0.00003		0.03283		0.02062		0.0085(1)	C	0.028(1)	C	880	N	61	N	0.00003	0.00003	0.00003
2-AMINO-4,6-DINITROTOLUENE	35572-78-2							---		---						120(2)	N	4.7(2)	N	4.7	4.7	4.7
2-NITROTOLUENE	88-72-2							---		---						1000	sat	370	N	370	370	370
3-NITROTOLUENE	99-08-1							---		---						1000	sat	370	N	370	370	370
4-AMINO-2,6-DINITROTOLUENE	19406-51-0							---		---						120(2)	N	4.7(2)	N	4.7	4.7	4.7
4-NITROTOLUENE	99-99-0							---		---						1000	sat	370	N	370	370	370
RDX	121-82-4							---		---						22	C	4.4	C	4.4	4.4	4.4
TETRYL	479-45-8							---		---						20000(2)	N	780(2)	N	780	780	780
NITROBENZENE	98-95-3	39		92		0.007		1.31		0.4876		0.028	N	0.34	N	110	N	20	N	0.007	0.007	0.007
HMX	2691-41-0							---		---						44000	N	3100	N	3100	3100	3100
LAUCKS HPLC METHOD																						
PICRAMIC ACID	96-91-3							---		---												
PICRIC ACID	88-89-1							---		---												
METALS																						
APPENDIX IX SW-846 METHOD 6020 ICP/MS																						
ANTIMONY	7440-36-0	31				0.3		0.1423		---		5.4	N	37	N	820	N	31	N	0.1423	0.3	0.1423
ARSENIC	7440-38-2	0.4		750		1		5.7		5.9		3.9	C	20	C	2.7	C	0.39	C	0.39	5.9	0.39
BARIUM	7440-39-3	5500		690000		82		1.04		---		1600	N	5900	N	100000	N	5400	N	1.04	82	1.04
BERYLLIUM	7440-41-7	0.1		1300		3		1.06		---		63	C	2300	N	2200	N	150	N	0.1	0.1	0.1
CADMIUM	7440-43-9	78		1800		0.4		0.00222		0.596		7.5	N	77	N	810	N	37	N	0.00222	0.4	0.00222
CHROMIUM	7440-47-3	390	3	270	3	2	3	0.4		26		38	C	120	C	450	C	210	C	0.4	2	0.4
COBALT	7440-48-4							0.14033		50						100000	N	4700	N	0.14033	50	0.14033
COPPER	7440-50-8							2.96		16		580	N	1700	N	76000	N	2900	N	2.96	16	2.96
LEAD	7439-92-1	400	4					0.05373		31		81		230		750	5	400	4	0.05373	31	0.05373
LITHIUM	743993-2															41000	N	1600	N	1600	1600	1600
MERCURY (SW-846 METHOD 7470A/747	7439-97-6	23		10	6	0.1		0.073		0.174		2.1	N	32	N	610	N	23	N	0.073	0.1	0.073
NICKEL	7440-02-0	1600		13000		7		13.6		16		950	N	2700	N	41000	N	1600	N	7	7	7
SELENIUM	7782-49-2	390				0.3		0.02765		---		5.2	N	53	N	10000	N	390	N	0.02765	0.3	0.02765
SILVER	7440-22-4	390				2		4.04		0.5		31	N	87	N	10000	N	390	N	2	0.5	0.5
STRONTIUM	7440-24-6															100000	max	47000	N	47000	47000	47000
THALLIUM	7440-28-0					0.04		0.05692		---		2.8	N	13	N	130	N	5.2	N	0.04	0.04	0.04
TIN	7440-31-5							7.62		---						100000	N	47000	N	7.62	47000	7.62
VANADIUM	7440-62-2	550				300		1.59		---						14000	N	550	N	1.59	300	1.59
ZINC	7440-66-6	23000				620		6.62		120		10000	N	10000	N	100000	N	23000	N	6.62	120	6.62
ALUMINUM	7429-90-5							---		---						100000	N	76000	N	76000	76000	76000
IRON	7439-89-6							---		---						100000	N	23000	N	23000	23000	23000
MAGNESIUM	7439-95-4							---		---												
MANGANESE	7439-96-5							---		---						32000	N	1800	N	1800	1800	1800
CALCIUM	7440-70-2							---		---												
POTASSIUM	7440-09-7							---		---												
SODIUM	7440-23-5							---		---												
TITANIUM	7440-32-6							---		---												
APPENDIX IX VOLATILE ORGANIC COMPOUNDS																						
SW-846 METHOD 8260B																						
1,1,1,2-TETRACHLOROETHANE	630-20-6							225		0.01089		0.05	C	0.79	C	7	C	3	C	0.05	0.01089	0.01089
1,1,1-TRICHLOROETHANE	71-55-6			1200		0.1		29.8		0.24685		1.9	N	35	N	1400	N	630	N	0.1	0.1	0.1
1,1,2,2-TETRACHLOROETHANE	79-34-5	3		0.6		0.0002		0.12722		0.02908		0.007	C	0.11	C	0.9	C	0.38	C	0.0002	0.0002	0.0002
1,1,2-TRICHLOROETHANE	79-00-5	11		1		0.0009		28.6		0.67351		0.03	C	0.3	C	1.9	C	0.84	C	0.0009	0.0009	0.0009
1,1-DICHLOROETHANE	75-34-3	7800		1300		1		20.1		0.000575		5.6	N	58	N	2100	N	590	N	1	0.000575	0.000575
1,1-DICHLOROETHENE	75-35-4	1		0.07		0.003		8.28		0.02327		0.058	C	0.058	C	0.12	C	0.054	C	0.003	0.003	0.003
1,2,3-TRICHLOROPROPANE	96-18-4							3.36		0.00835						0.0031	C	0.0014	C	0.0014	0.0014	0.0014
1,2-DIBROMO-3-CHLOROPROPANE	96-12-8							0.03518		0.01998						4	C	0.45	C	0.03518	0.01998	0.01998
1,2-DIBROMOETHANE	106-93-4							1.23		0.01237						0.048	C	0.0069	C	0.0069	0.0069	0.0069
1,2-DICHLOROETHANE	107-06-2	7		0.4		0.001		21.2		0.05418		0.024	C	0.15	C	0.76	C	0.35	C	0.001	0.001	0.001
1,2-DICHLOROPROPANE	78-87-5	9		15		0.001		32.7		0.35161		0.03	C	0.25	C	0.77	C	0.35	C	0.001	0.001	0.001
2-BUTANONE	78-93-3							89.6		0.13696		10	N	260	N	28000	N	7300	N	10	0.13696	0.13696
2-HEXANONE	591-78-6							12.6		1.01						82000(2)	N	3100(2)	N	12.6	1.01	1.01
3-CHLOROPROPENE	107-05-1							---		---						43000	N	3000	N	3000	3000	3000
4-METHYL-2-PENTANONE	108-10-1							443		0.54437						2900	N	790	N	443	0.54437	0.54437
ACETONE	67-64-1	7800		100000		0.8		2.5		0.45337		3.1	N	41	N	6200	N	1600	N	0.8	0.45337	0.45337
ACROLEIN	107-02-8							5.27		1.44E-05		0.00023	N	0.22	N	0.34	N	0.1	N	0.00023	0.0000144	0.0000144
ACRYLONITRILE	107-13-1							---		---						0.51	C	0.21	C	0.21	0.21	0.21
BENZENE	71-43-2	22		0.8		0.002		0.25462		0.14157		0.34	C	0.67	C	1.5	C	0.65	C	0.002	0.002	0.002
BROMODICHLOROMETHANE	75-27-4	10		3000		0.03		0.53978		0.00113		0.63	C	0.63	C	2.4	C	1	C	0.03	0.00113	0.00113

APPENDIX B
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NSWC Crane, Crane, Indiana

PARAMETER	CAS	EPA GENERIC SSLs INGESTION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs INHALATION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs MIGRATION TO GW (mg/kg) DAF=1	FOOTNOTE	REGION 5 ECO DQLs SOIL (mg/kg)	FOOTNOTE	REGION 5 ECO DQLs SEDIMENT (mg/kg)	FOOTNOTE	IDEM DEFAULT RESIDENTIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	IDEM DEFAULT INDUSTRIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR INDUSTRIAL LAND USE (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR RESIDENTIAL LAND USE (mg/kg)	FOOTNOTE	MINIMUM CRITERIA VALUE SOIL (mg/kg)	MINIMUM CRITERIA VALUE SEDIMENT (mg/kg)	MINIMUM CRITERIA VALUE SOIL AND SEDIMENT (mg/kg)
BROMOFORM	75-25-2	81		53		0.04		15.9		0.99627		0.75	C	2.7	C	310	C	62	C	0.04	0.04	0.04
BROMOMETHANE	74-83-9	110		10		0.01		0.23516		0.94674		0.052	N	0.7	N	13	N	3.9	N	0.01	0.01	0.01
CARBON DISULFIDE	75-15-0	7800		720		2		0.09412		0.13397		10	N	82	N	720	N	360	N	0.09412	0.13397	0.09412
CARBON TETRACHLORIDE	56-23-5	5		0.3		0.003		2.98		0.03573		0.066	C	0.29	C	0.53	C	0.24	C	0.003	0.003	0.003
CHLOROBENZENE	108-90-7	1600		130		0.07		13.1		0.06194		1.3	N	27	N	540	N	150	N	0.07	0.06194	0.06194
CHLORODIBROMOMETHANE	124-48-1	8		1300		0.02		---		---						2.7	C	1.1	C	0.02	0.02	0.02
CHLOROETHANE	75-00-3							---		58.6						6.5	C	3	C	3	3	3
CHLOROFORM	67-66-3	100		0.3		0.03		1.19		0.027		0.59	C	1.2	C	0.52	C	0.24	C	0.03	0.027	0.027
CHLOROMETHANE	74-87-3							10.4		7.85E-05						2.7	C	1.2	C	1.2	0.0000785	0.0000785
CIS-1,2-DICHLOROETHENE	156-59-2	780		1200		0.02		0.78373	7	0.20894	7	0.4	N	5.8	N	150	N	43	N	0.02	0.02	0.02
CHLOROPRENE	126-99-8							---		---						12	N	3.6	N	3.6	3.6	3.6
CIS-1,3-DICHLOROPROPENE	10061-01-5	4	37	0.1	37	0.0002	37	0.39786		0.00296		0.04(8)	C	0.2(8)	C	1.6	C	0.7	C	0.0002	0.0002	0.0002
DIBROMOMETHANE	74-95-3							---		---						240	N	67	N	67	67	67
DICHLORODIFLUOROMETHANE	75-71-8							39.5		0.00133						310	N	94	N	39.5	0.00133	0.00133
ETHYL METHACRYLATE	97-63-2							30		0.000602						140	sat	140	sat	30	0.000602	0.000602
ETHYLBENZENE	100-41-4	7800		400		0.7		5.16		0.0001		13	N	200	N	230	N	230	N	0.7	0.0001	0.0001
METHACRYLONITRILE	126-98-7							0.05705		2.97E-05						8.8	N	2.1	N	0.05705	0.0000297	0.0000297
METHYL IODIDE	74-88-4									---												
METHYL METHACRYLATE	80-62-6							984		0.16756						2700	N	2200	N	984	0.16756	0.16756
METHYLENE CHLORIDE	75-09-2	85		13		0.001		4.05		1.26		0.023	C	1.8	C	21	C	8.9	C	0.001	0.001	0.001
STYRENE	100-42-5	16000		1500		0.2		4.69		0.44496		3.5	N	720	N	1700	sat	1700	sat	0.2	0.2	0.2
TETRACHLOROETHENE	127-18-4	12		11		0.003		9.92		0.19583		0.058	C	0.64	C	19	C	5.7	C	0.003	0.003	0.003
TOLUENE	108-88-3	16000		650		0.6		5.45		52.5		12	N	240	N	520	sat	520	sat	0.6	0.6	0.6
TOTAL XYLENES	1330-20-7	160000	61	410	61	9	61	10		1.88		190	N	410	N	210	sat	210	sat	9	1.88	1.88
TRANS-1,2-DICHLOROETHENE	156-60-5	1600		3100		0.03		0.78373		0.20894		0.68	N	14	N	210	N	63	N	0.03	0.03	0.03
TRANS-1,3-DICHLOROPROPENE	10061-02-6	4	37	0.1	37	0.0002	37	0.39786		0.00296		0.04(8)	C	0.2(8)	C	1.6	C	0.7	C	0.0002	0.0002	0.0002
TRANS-1,4-DICHLORO-2-BUTENE	110-57-6							---		---						0.018(9)	C	0.0079(9)	C	0.0079	0.0079	0.0079
TRICHLOROETHENE	79-01-6	58		5		0.003		12.4		0.17956		0.057	C	3	C	6.1	C	2.8	C	0.003	0.003	0.003
TRICHLOROFLUOROMETHANE	75-69-4							16.4		0.00307						2000	N	390	N	16.4	0.00307	0.00307
VINYL ACETATE	108-05-4	78000		1000		8		12.7		0.01295		2.3	N	430	N	1400	N	430	N	2.3	0.01295	0.01295
VINYL CHLORIDE	75-01-4	0.3		0.03		0.0007		0.64614		0.002		0.013	C	0.013	C	0.83	C	0.15	C	0.0007	0.0007	0.0007
SW-846 METHOD 8015B																						
1,4-DIOXANE	123-91-1							2.05		5.43E-06						220	C	44	C	2.05	5.4E-06	5.4E-06
ACETONITRILE	75-05-8							1.37		0.13905						1700	N	270	N	1.37	0.13905	0.13905
ISOBUTANOL	78-83-1							20.8		3.35						40000	N	13000	N	20.8	3.35	3.35
PROPIONITRILE	107-12-0							0.04983		0.11466										0.04983	0.11466	0.04983
APPENDIX IX SEMIVOLATILE ORGANIC COMPOUNDS																						
SW-846 METHOD 8270C																						
1,2,4,5-TETRACHLOROBENZENE	95-94-3							2.02		20.9						260	N	18	N	2.02	18	2.02
1,2,4-TRICHLOROBENZENE	120-82-1	780		3200		0.3		11.1		11.7		5.3	N	77	N	3000	N	650	N	0.3	0.3	0.3
1,2-DICHLOROBENZENE	95-50-1	7000		560		0.9		2.96		0.23132		17	N	270	N	370	sat	370	sat	0.9	0.23132	0.23132
1,3-DICHLOROBENZENE	541-73-1							37.7		3.01		0.13	N	1.8	N	52	N	13	N	0.13	0.13	0.13
1,4-DICHLOROBENZENE	106-46-7	27				0.1		---		---		2.2	C	3.4	C	8.1	C	3.4	C	0.1	0.1	0.1
1,4-NAPHTHOQUINONE	130-15-4							---		---												
1,4-PHENYLENEDIAMINE	106-50-3							---		---						100000	max	12000	N	12000	12000	12000
1-NAPHTHYLAMINE	134-32-7							---		---												
2,3,4,6-TETRACHLOROPHENOL	58-90-2							0.19878		1.51						26000	N	1800	N	0.19878	1.51	0.19878
2,4,5-TRICHLOROPHENOL	95-95-4	7800				14		14.1		0.08556		250	N	680	N	88000	N	6100	N	14	0.08556	0.08556
2,4,6-TRICHLOROPHENOL	88-06-2	58		200		0.008		9.94		0.08484		1.5	C	5	C	220	C	44	C	0.008	0.008	0.008
2,4-DICHLOROPHENOL	120-83-2	230				0.05		87.5		0.13363		1.1	N	3	N	2600	N	180	N	0.05	0.05	0.05
2,4-DIMETHYLPHENOL	105-67-9	1600				0.4		0.01		0.30453		9	N	25	N	18000	N	1200	N	0.01	0.30453	0.01
2,4-DINITROPHENOL	51-28-5	160				0.01		0.06086		0.00133		0.29	N	0.82	N	1800	N	120	N	0.01	0.00133	0.00133
2,6-DICHLOROPHENOL	87-65-0							---		---												
2-ACETYLAMINOFLUORENE	53-96-3							0.59634		0.01532										0.59634	0.01532	0.01532
2-CHLORONAPHTHALENE	91-58-7							0.01218		0.41723						27000	N	3900	N	0.01218	0.41723	0.01218
2-CHLOROPHENOL	95-57-8	390		53000		0.2		0.24266		0.0117		0.75	N	10	N	240	N	63	N	0.2	0.0117	0.0117
2-METHYLPHENOL	95-48-7	3900				0.8		40.4		0.000826		14	N	39	N	44000	N	3100	N	0.8	0.000826	0.000826
2-NAPHTHYLAMINE	91-59-8							---		---												
2-NITROANILINE	88-74-4							74.1		0.000222						50	N	3.5	N	3.5	0.000222	0.000222
2-NITROPHENOL	88-75-5							1.6		0.00777						7000(10)	N	490(10)	N	1.6	0.00777	0.00777
2-PICOLINE	109-06-8							9.9		0.75305										9.9	0.75305	0.75305
3,3'-DICHLOROBENZIDINE	91-94-1	1				0.0003		0.64636		0.02822		0.062	C	0.21	C	5.5	C	1.1	C	0.0003	0.0003	0.0003
3,3'-DIMETHYLBENZIDINE	119-93-7							---		---						0.27	C	0.053	C	0.053	0.053	0.053
3-METHYLCHOLANTHRENE	56-49-5							0.07794		8190										0.07794	8190	0.07794
3-METHYLPHENOL	108-39-4							3.49		0.000845						44000	N	3100	N	3.49	0.000845	0.000845
3-NITROANILINE	99-09-2							---		---						50	N	3.5	N	3.5	3.5	3.5
4,6-DINITRO-2-METHYLPHENOL	534-52-1							0.14408		0.01038										0.14408	0.01038	0.01038
4-AMINOBIPHENYL	92-67-1							---		---												
4-BROMOPHENYL PHENYL ETHER	101-55-3							---		1.55											1.55	1.55
4-CHLORO-3-METHYLPHENOL	59-50-7							7.95		0.38818										7.95	0.38818	0.38818
[illegible]LOROANILINE	106-47-8	310				0.03		1.1		[illegible]		0.97	N	2.7	N	3500	N	240	N	0.03	0.03	0.03

APPENDIX B
Table 2
Tabular Presentation of Soil and Sediment RBSLs for RBTL Selection
NSWC Crane, Crane, Indiana

PARAMETER	CAS	EPA GENERIC SSLs INGESTION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs INHALATION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs MIGRATION TO GW (mg/kg) DAF=1	FOOTNOTE	REGION 5 ECO DQLs SOIL (mg/kg)	FOOTNOTE	REGION 5 ECO DQLs SEDIMENT (mg/kg)	FOOTNOTE	IDEM DEFAULT RESIDENTIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	IDEM DEFAULT INDUSTRIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR INDUSTRIAL LAND USE (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR RESIDENTIAL LAND USE (mg/kg)	FOOTNOTE	MINIMUM CRITERIA VALUE SOIL (mg/kg)	MINIMUM CRITERIA VALUE SEDIMENT (mg/kg)	MINIMUM CRITERIA VALUE SOIL AND SEDIMENT (mg/kg)
4-CHLOROPHENYL PHENYL ETHER	7005-72-3									---												
4-METHYLPHENOL	106-44-5							163		0.000808						4400	N	310	N	163	0.000808	0.000808
4-NITROANILINE	100-01-6							---		---						50	N	3.5	N	3.5	3.5	3.5
4-NITROPHENOL	100-02-7							5.12		0.00778						7000		490		5.12	0.00778	0.00778
4-NITROQUINOLINE-1-OXIDE	56-57-5							---		---												
5-NITRO-O-TOLUIDINE	99-55-8							---		---						75	C	15	C	15	15	15
7,12-DIMETHYLBENZ(A)ANTHRACENE	57-97-6							16.3		66.4										16.3	66.4	16.3
A,A-DIMETHYLPHENETHYLAMINE	122-09-8							---		---						880	N	61	N	61	61	61
ACETOPHENONE	98-86-2							300		0.246						1.6	N	0.49	N	0.49	0.246	0.246
ANILINE	62-53-3							0.05678		3.38E-05						430	C	85	C	0.05678	0.0000338	0.0000338
ARAMITE	140-57-8							166		1.11E-06						99	C	19	C	19	0.00000111	0.00000111
BENZYL ALCOHOL	100-51-6							65.8		0.03394						100000	N	18000	N	85.8	0.03394	0.03394
BIS(2-CHLOROETHOXY)METHANE	111-91-1							0.30209		0.34971										0.30209	0.34971	0.30209
BIS(2-CHLOROETHYL)ETHER	111-44-4	0.6		0.2		0.00002		23.7		0.21196		0.0007	C	0.012	C	0.62	C	0.21	C	0.00002	0.00002	0.00002
BIS(2-CHLOROISOPROPYL)ETHER	39638-32-9							---		---												
BIS(2-ETHYLHEXYL)PHTHALATE	117-81-7	46		31000		180		0.92594		0.182		300	C	980	C	180	C	35	C	0.92594	0.182	0.182
BUTYL BENZYL PHTHALATE	85-68-7	16000		930		810		0.23889		4.19		930	N	930	N	100000	N	12000	N	0.23889	4.19	0.23889
CHLOROBENZILATE	510-15-6							5.05		0.86029						9.1	C	1.8	C	1.8	0.86029	0.86029
DIALLATE	2303-16-4							0.45214		0.00151						40	C	8	C	0.45214	0.00151	0.00151
DIBENZOFURAN	132-64-9							---		1.52						5100	N	290	N	290	1.52	1.52
DIETHYL PHTHALATE	84-66-2	63000		2000		23		24.8		0.00804		450	N	1300	N	100000	max	49000	N	23	0.00804	0.00804
DIMETHYL PHTHALATE	131-11-3							734		0.02495		1400	sat	1400	sat	100000	max	100000	max	734	0.02495	0.02495
DI-N-BUTYL PHTHALATE	84-74-2	7800		2300		270		0.14979		0.1105		2000	max	2000	max	88000	N	6100	N	0.14979	0.1105	0.1105
DI-N-OCTYL PHTHALATE	117-84-0	1600		10000		10000		709		40.6		2000	max	2000	max	10000	N	1200	N	709	40.6	40.6
DIPHENYLAMINE	122-39-4							1.01		0.0346						22000	N	1500	N	1.01	0.0346	0.0346
ETHYL METHANE SULFONATE	62-50-0							---		---												
HEXACHLOROBENZENE	118-74-1	0.4		1		0.1		0.19878		0.02		2.2	C	3.9	C	1.5	C	0.3	C	0.1	0.02	0.02
HEXACHLOROBUTADIENE	87-68-3	8		8		0.1		0.03976		1.38		16	C	44	C	32	C	6.2	C	0.03976	0.1	0.03976
HEXACHLOROCYCLOPENTADIENE	77-47-4	550		10		20		0.75537		0.90074		400	N	2000	N	5900	N	420	N	0.75537	0.90074	0.75537
HEXACHLOROETHANE	67-72-1	46		55		0.02		0.59634		2.23		2.8	C	7.7	C	180	C	35	C	0.02	0.02	0.02
HEXACHLOROPROPENE	1888-71-7							---		0.0002											0.0002	0.0002
ISODRIN	465-73-6							0.00332		0.05516										0.00332	0.05516	0.00332
ISOPHORONE	78-59-1	670		4600		0.03		139		0.4223		5.3	C	18	C	2600	C	510	C	0.03	0.03	0.03
ISOSAFROLE	120-58-1							9.94		0.00412										9.94	0.00412	0.00412
KEPONE	143-50-0							0.03272		0.00331						0.14	C	0.027	C	0.027	0.00331	0.00331
METHAPYRILENE	91-80-5							2.78		1.44E-05										2.78	1.44E-05	1.44E-05
METHYL METHANE SULFONATE	66-27-3							0.31549		4.74E-06										0.31549	4.74E-06	4.74E-06
N-NITROSODIETHYLAMINE	55-18-5							0.06933		0.02277						0.016	C	0.0032	C	0.0032	0.0032	0.0032
N-NITROSODIMETHYLAMINE	62-75-9							3.21E-05		2.75E-06						0.048	C	0.0095	C	3.2E-05	2.8E-06	2.8E-06
N-NITROSODI-N-BUTYLAMINE	924-16-3							0.26707		0.77204						0.061	C	0.024	C	0.024	0.024	0.024
N-NITROSO-DI-N-PROPYLAMINE	621-64-7	0.09				0.000002		0.54368		0.000217		0.0006	C	0.002	C	0.35	C	0.069	C	2.0E-06	2.0E-06	2.0E-06
N-NITROSOMETHYLETHYLAMINE	10595-95-6							0.00166		4.85E-06						0.11	C	0.022	C	0.00166	4.9E-06	4.9E-06
N-NITROSOMORPHOLINE	59-89-2							0.07057		3.70E-06										0.07057	3.7E-06	3.7E-06
N-NITROSOPIPERIDINE	100-75-4							0.00665		2.26E-05										0.00665	2.3E-05	2.3E-05
N-NITROSOPYRROLIDINE	930-55-2							0.01258		9.08E-07						1.2	C	0.23	C	0.01258	9.1E-07	9.1E-07
O,O,O-TRIETHYL PHOSPHOROTHIOATE	126-68-1							---		---												
O-TOLUIDINE	95-53-4							---		---						10	C	2	C	2	2	2
P-(DIMETHYLAMINO)AZOBENZENE	60-11-7							---		---												
PENTACHLOROBENZENE	608-93-5							0.49695		1.26						700	N	49	N	0.49695	1.26	0.49695
PENTACHLOROETHANE	76-01-7							10.7		0.68918										10.7	0.68918	0.68918
PENTACHLORONITROBENZENE	82-68-8							7.09		18.2						9.5	C	1.9	C	1.9	1.9	1.9
PHENACETIN	62-44-2							11.7		0.00225										11.7	0.00225	0.00225
PHENOL	108-95-2	47000				5		120		0.02726		110	N	320	N	100000	N	37000	N	5	0.02726	0.02726
PRONAMIDE	23950-58-5							0.0136		0.0016						66000	N	4600	N	0.0136	0.0016	0.0016
PYRIDINE	110-86-1							1.03		0.10617						880	N	61	N	1.03	0.10617	0.10617
SAFROLE	94-59-7							0.40398		0.16486										0.40398	0.16486	0.16486
8270C-SELECTIVE ION MONITORING																						
2-METHYLNAPHTHALENE	91-57-6							3.24		0.0202		0.7	11	170	11	190	11	56	11	0.7	0.0202	0.0202
ACENAPHTHENE	83-32-9	4700				29		682		0.00671		130	N	1200	N	38000	N	3700	N	29	0.00671	0.00671
ACENAPHTHYLENE	208-96-8							682		0.00587		130	12	1200	12	38000	12	3700	12	130	0.00587	0.00587
ANTHRACENE	120-12-7	23000				590		1480		0.0469		51	N	51	N	100000	N	22000	N	51	0.0469	0.0469
BENZO(A)ANTHRACENE	56-55-3	0.9				0.08		5.21		0.0317		5	C	15	C	2.9	C	0.62	C	0.08	0.0317	0.0317
BENZO(A)PYRENE	50-32-8	0.09				0.4		1.52		0.0319		0.5	C	1.5	C	0.29	C	0.062	C	0.062	0.0319	0.0319
BENZO(B)FLUORANTHENE	205-99-2	0.9				0.2		59.8		10.4		5	C	15	C	2.9	C	0.62	C	0.2	0.2	0.2
BENZO(G,H,I)PERYLENE	191-24-2							119		0.17		570	13	570	13	54000	13	2300	13	119	0.17	0.17
BENZO(K)FLUORANTHENE	207-08-9	9				2		148		0.24		39	C	39	C	29	C	6.2	C	2	0.24	0.24
CHRYSENE	218-01-9	88				8		4.73		0.0571		25	C	25	C	290	C	62	C	4.73	0.0571	0.0571
DIBENZO(A,H)ANTHRACENE	53-70-3	0.09				0.08		18.4		0.00622		0.5	C	1.5	C	0.29	C	0.062	C	0.062	0.00622	0.00622
FLUORANTHENE	206-44-0	3100				210		122		0.1113		880	N	880	N	30000	N	2300	N	122	0.1113	0.1113
FLUORENE	86-73-7	3100				28		122		0.0212		170	N	1100	N	33000	N	2600	N	28	0.0212	0.0212

APPENDIX B
Table 2
Tabular Presentation of Soil and Sediment RBSLs for RBTL Selection
NSWC Crane, Crane, Indiana

PARAMETER	CAS	EPA GENERIC SSLs INGESTION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs INHALATION (mg/kg)	FOOTNOTE	EPA GENERIC SSLs MIGRATION TO GW (mg/kg) DAF=1	FOOTNOTE	REGION 5 ECO DOLs SOIL (mg/kg)	FOOTNOTE	REGION 5 ECO DOLs SEDIMENT (mg/kg)	FOOTNOTE	IDEM DEFAULT RESIDENTIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	IDEM DEFAULT INDUSTRIAL SOIL CLOSURE LEVELS (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR INDUSTRIAL LAND USE (mg/kg)	FOOTNOTE	REGION 9 SOIL PRGs FOR RESIDENTIAL LAND USE (mg/kg)	FOOTNOTE	MINIMUM CRITERIA VALUE SOIL (mg/kg)	MINIMUM CRITERIA VALUE SEDIMENT (mg/kg)	MINIMUM CRITERIA VALUE SOIL AND SEDIMENT (mg/kg)
INDENO(1,2,3-CD)PYRENE	193-39-5	0.9				0.7		109		0.2		3.1	C	3.1	C	2.9	C	0.62	C	0.62	0.2	0.2
NAPHTHALENE	91-20-3	3100				4		0.09939		0.0346		0.7	N	170	N	190	N	56	N	0.09939	0.0346	0.0346
PHENANTHRENE	85-01-8							45.7		0.0419		570	13	570	13	54000	13	2300	13	45.7	0.0419	0.0419
PYRENE	129-00-0	2300				210		78.5		0.053		570	N	570	N	54000	N	2300	N	78.5	0.053	0.053
APPENDIX IX ORGANOCHLORINE PCBs																						
SW-846 METHOD 8082																						
AROCLOR-1016	12674-11-2	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	29	C	3.9	N	0.67971	0.0341	0.0341
AROCLOR-1221	11104-28-2	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	1	C	0.22	C	0.22	0.0341	0.0341
AROCLOR-1232	11141-16-5	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	1	C	0.22	C	0.22	0.0341	0.0341
AROCLOR-1242	53469-21-9	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	1	C	0.22	C	0.22	0.0341	0.0341
AROCLOR-1248	12672-29-6	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	1	C	0.22	C	0.22	0.0341	0.0341
AROCLOR-1254	11097-69-1	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	1	C	0.22	C	0.22	0.0341	0.0341
AROCLOR-1260	11096-82-5	1	14					0.67971	14	0.0341	14	1.8(14)	C	5.3(14)	C	1	C	0.22	C	0.22	0.0341	0.0341
HERBICIDES																						
SW-846 METHOD 8151A																						
HEXACHLOROPHENE	70-30-4							0.19878		231						260	N	18	N	0.19878	18	0.19878
PENTACHLOROPHENOL	87-86-5	3	58			0.001		0.11927		30.1		0.028	C	0.66	C	11	C	3	C	0.001	0.001	0.001
DIOXINS/FURANS																						
SW-846 METHOD 8290																						
1,2,3,4,6,7,8,9-OCDD	3268-87-9							1.99E-07		3.3E-06						0.27	C	0.039	C	2.0E-07	3.3E-06	2.0E-07
1,2,3,4,6,7,8,9-OCDF	39001-02-0							3.86E-05		1.29E-08						0.27	C	0.039	C	3.9E-05	1.3E-08	1.3E-08
1,2,3,4,6,7,8-HPCDD	35822-46-9							1.99E-07		3.3E-06						0.0027	C	0.00039	C	2.0E-07	3.3E-06	2.0E-07
1,2,3,4,6,7,8-HPCDF	67562-39-4							3.86E-05		1.29E-08						0.0027	C	0.00039	C	3.9E-05	1.3E-08	1.3E-08
1,2,3,4,7,8,9-HPCDF	55673-89-7							3.86E-05		1.29E-08						0.0027	C	0.00039	C	3.9E-05	1.3E-08	1.3E-08
1,2,3,4,7,8-HXCDD	39227-28-6							1.99E-07		3.3E-06						0.00027	C	0.000039	C	2.0E-07	3.3E-06	2.0E-07
1,2,3,4,7,8-HXCDF	70648-26-9							3.86E-05		1.29E-08						0.00027	C	0.000039	C	3.9E-05	1.3E-08	1.3E-08
1,2,3,6,7,8-HXCDD	57653-85-7							1.99E-07		3.3E-06						0.00027	C	0.000039	C	2.0E-07	3.3E-06	2.0E-07
1,2,3,6,7,8-HXCDF	57117-44-9							3.86E-05		1.29E-08						0.00027	C	0.000039	C	3.9E-05	1.3E-08	1.3E-08
1,2,3,7,8,9-HXCDD	19408-74-3							1.99E-07		3.3E-06						0.00027	C	0.000039	C	2.0E-07	3.3E-06	2.0E-07
1,2,3,7,8,9-HXCDF	72918-21-9							3.86E-05		1.29E-08						0.00027	C	0.000039	C	3.9E-05	1.3E-08	1.3E-08
1,2,3,7,8-PECDD	40321-76-4							1.99E-07		3.3E-06						0.000027	C	0.0000039	C	2.0E-07	3.3E-06	2.0E-07
1,2,3,7,8-PECDF	57117-41-6							3.86E-05		1.29E-08						0.00054	C	0.000078	C	3.9E-05	1.3E-08	1.3E-08
2,3,4,6,7,8-HXCDF	60851-34-5							3.86E-05		1.29E-08						0.00027	C	0.000039	C	3.9E-05	1.3E-08	1.3E-08
2,3,4,7,8-PECDF	57117-31-4							3.86E-05		1.29E-08						0.000054	C	0.0000078	C	7.8E-06	1.3E-08	1.3E-08
2,3,7,8-TCDD	1746-01-6							1.99E-07		3.3E-06						0.000027	C	0.0000039	C	2.0E-07	3.3E-06	2.0E-07
2,3,7,8-TCDF	51207-31-9							3.86E-05		1.29E-08						0.00027	C	0.000039	C	3.9E-05	1.3E-08	1.3E-08
TOTAL HPCDD								---		---												
TOTAL HPCDF								---		---												
TOTAL HXCDD								---		---												
TOTAL HXCDF								---		---												
TOTAL PECDD								---		---												
TOTAL PECDF								---		---												
TOTAL TCDD								---		---												
TOTAL TCDF								---		---												
MISCELLANEOUS PARAMETERS-ALL ROUNDS																						
TOTAL ORGANIC CARBON SW-846 METHOD 9060	TTNUS003							---		---												
PERCHLORATE-EPA METHOD 314	14797-73-0							---		---												
NITRATE + NITRITE AS NITRATE-EPA 353.2	14797-55-8							---		---												
HARDNESS STANDARD METHOD A28323408	TTNUS022							---		---												
PH-SW-846 METHOD 9045C	TTNUS002							---		---												
TOTAL SUSPENDED SOLIDS-EPA METHOD 160.2	TTNUS044							---		---												
CEC SW-846 METHOD 9081	14797-65-0							---		---												
CYANIDE- METHOD 9012A	57-12-5	1600				2		1.33		0.0001		150	N	410	N	35	N	11	N	1.33	0.0001	0.0001
AMMONIA-EPA 350.1	7664-41-7							---		---												

1 Dinitrotoluene mixture
2 EPA Region 3 risk-based concentration.
3 Hexavalent Chromium
4 OSWER soil screening level for residential land use (USEPA, 1994).
5 Recommended screening level for commercial/industrial sites (TRW Group, 1999).
6 Value is for elemental, volatile mercury.
7 trans-1,2-dichloroethene
8 1,3-dichloropropene.
9 Value for 1,4-dichloro-2-butene presented.
10 Value for 4-nitrophenol presented.
11 Value for naphthalene presented.
12 Value for acenaphthene presented.
13 Value for pyrene presented.
14 Total for all Aroclor congeners.

APPENDIX C

HUMAN HEALTH RISK ASSESSMENT METHODOLOGY

APPENDIX C
HUMAN HEALTH RISK ASSESSMENT METHODOLOGY

This section of the QAPP outlines the general methodologies and procedures that will be used to conduct the baseline human health risk assessment for Mine Fill A (SWMU 12), Mine Fill B (SWMU 13), the Cast High Explosive Fill/B146 Incinerator (SWMU 16), and the Pyrotechnic Test Areas (SWMU 19). The objective of the risk assessment is to determine whether detected concentrations of chemicals within the study area pose a significant threat to potential human receptors under current and/or future land use. The potential risks to human receptors will be estimated based on the assumption that no actions are taken to control contaminant releases.

The following current United States Environmental Protection Agency (U.S. EPA) and the state of Indiana Department of Environmental Management (IDEM) risk assessment guidance documents were used to develop the framework for the baseline human health risk assessment:

- Risk Assessment Guidance for Superfund: Volume I, Human Health Evaluation Manual (Part A). (U.S. EPA, 1989).

- Human Health Evaluation Manual, Supplemental Guidance: Standard Default Exposure Factors. Washington, D.C. OSWER Directive 9285.6-03 (U.S. EPA, 1991a)

- Supplemental Guidance to RAGS: Calculating the Concentration Term. OSWER Publication No. 9285.7-081 (U.S. EPA, 1992c)

- Distribution of Preliminary Review Draft: Superfund's Standard Default Exposure Factors for the Central Tendency and Reasonable Maximum Exposure. Office of Solid Waste and Emergency Response, Washington, D.C. (U.S. EPA, 1993a).

- Soil Screening Guidance: Technical Background Document. Office of Solid Waste and Emergency Response, Washington, D.C. EPA/540/R-95/128 (U.S. EPA, 1996a).

- Exposure Factors Handbook. Office of Health and Environmental Assessment, Washington, D.C. EPA/600/P-95/002Fa (U.S. EPA, 1997a).

- Risk Assessment Guidance for Superfund: Volume 1 - Human Health Evaluation Manual (Part D, Standardized Planning, Reporting, and Review of Superfund Risk Assessments) (U.S. EPA, 1998a).

- Risk Assessment Guidance for Superfund, Volume I: Human Health Evaluation Manual (Part E, Supplemental Guidance for Dermal Risk Assessment), Interim Guidance. Office of Emergency and Remedial Response, U.S. Environmental Protection Agency, Washington, D.C. 20460, Undated (received courtesy of U.S. EPA Region I, June 2000)

- Navy Interim Final Policy on the Use of Background Chemical Levels. Department of the Navy, September 2000.

- Conducting Human Health Risk Assessments under the Environmental Restoration Program. Department of the Navy, February 2001.

- RISC (Risk-Integrated System of Closure). Indiana Department of Environmental Management, Office of Environmental Response (February 2001).

A baseline human health risk assessment consists of five components: data evaluation, exposure assessment, toxicity assessment, risk characterization, and uncertainty analysis. Section 1 of this QAPP contains an overview of the methodologies to be followed for the five components of the risk assessment.

Three major aspects of chemical contamination and environmental fate and transport must be considered to evaluate potential risks: Contaminants with toxic characteristics must be found in environmental media and must be released by either natural processes or by human action; potential exposure points must exist; and human receptors must be present at the point of exposure. Risk is a function of both toxicity and exposure. If any one of the factors listed above is absent for a site, the exposure route is regarded as incomplete, and no potential risks will be considered to exist for human receptors.

C.1 DATA EVALUATION

Data evaluation, the first component of a baseline human health risk assessment, is a medium-specific task involving the compilation and evaluation of analytical data. The main objective of the data evaluation is to develop a medium-specific list of chemicals of potential concern (COPCs), which will be used to quantitatively determine potential human health risks for site media.

C.1.1 Data Usability

Data from samples collected for the proposed field investigations described in Section 3 of this QAPP will be used to assess risks to potential human receptors. All analytical data used in the quantitative estimation of potential risks will be subjected to data validation. A discussion of the data validation protocol to be followed is provided in the Section 8 of this QAPP. A Data Evaluation (see Sections 8 and 11) will be generated for the proposed field investigation data. Precision, accuracy, representativeness, completeness, and comparability will be evaluated and a brief summary of the results of the data validation will be documented. Additionally, the "data usability" evaluation worksheet suggested by U.S. EPA RAGS Part D (U.S. EPA 1998a) will be completed as a part of the data evaluation.

Fixed-base analytical results only from the Target Analyte Lists for the proposed field investigations and U.S.-EPA-approved historic data will be used in the quantitative risk evaluation. Typically, unfiltered results for ground water and surface water are used to assess risks associated with these media. Filtered ground water results will be used for the risk assessment if the associated unfiltered ground water samples are found to be turbid. Field measurements, data regarded as unreliable (i.e., qualified as "R" during the data validation process), historic data considered not acceptable by the U.S. EPA, and results of tentatively identified compound (TIC) analyses will not be used in the quantitative risk assessment. However, these data may be used to substantiate the conclusions of the quantitative risk analysis.

A discussion of the historical data for SWMUs 12, 13, 16, and 19 is provided in Section 1.3.6. Historical data from samples collected in 1997 and 1998 were used for an initial risk characterization of SWMUs 12 and 13, and the 1997 and 1998 data for SWMUs 12, 13, and 16 will be used in the quantitative risk analysis. Sections 1.3.6.1 and 1.3.6.2 contain a discussion of historical information for SWMUs 12 and 13, respectively. The 1997/1998 data provide a detailed description of post-remediation conditions, especially for the explosives and metals used at these SWMUs. However, there is uncertainty associated with the use of some of the historical data because the quality of the data has not been adequately documented and the data may not have been formally validated. The data packages (including the raw data) cannot be obtained to ascertain the level of quality associated with the data or to independently validate the data. Historical data deemed acceptable will be used in conjunction with the data obtained from the investigation described in this QAPP. The proposed field investigations were developed to be comprehensive (i.e., locations sampled historically, as well as data gap locations, were included); therefore, the uncertainty associated with the use of historical data in the quantitative risk assessment will be reduced.

C.1.2 Selection of Chemicals of Potential Concern

The selection of COPCs is a qualitative screening process used to limit the number of chemicals and exposure routes quantitatively evaluated in the baseline human health risk assessment to those site-related constituents that dominate overall potential risks. Screening by risk-based concentrations and basewide background levels will be used to focus the risk assessment on meaningful chemicals and exposure routes.

In general, a chemical will be selected as a COPC and retained for further quantitative risk evaluation if the maximum concentration detected in a sampled medium exceeds the lowest screening level and the chemical is determined to be present at concentrations exceeding background (only naturally occurring inorganics are eliminated as COPCs based on background comparisons). Chemicals eliminated from further evaluation at this time are assumed to present minimal risks to potential human receptors.

C.1.2.1 COPC Screening Levels

Several types of screening levels will be used to identify COPCs for SWMUs 12, 13, 16, and 19. Risk-based screening concentrations based on U.S. EPA Region 9 [referred to as preliminary remediation goals (PRGs)] (U.S. EPA, Region IX, 2000) will be used, as well as other IDEM (IDEM, 2001) and U.S. EPA criteria. The risk-based U.S. EPA Region 9 screening concentrations will correspond to a systemic Hazard Quotient of 0.1 (for noncarcinogens) or a lifetime cancer risk of 1E-6 (for carcinogens). It should be noted that the EPA Region 9 PRGs for noncarcinogens are based on a Hazard Index of 1. The Region 9 PRG values for noncarcinogens will be multiplied by 0.1 to account for potential cumulative effects of several chemicals affecting the same target area on or producing the same adverse noncarcinogenic health effect. The screening levels to be used for each medium in the risk assessment are briefly discussed below.

Soil/Sediment

The following criteria will be used to select COPCs for surface and subsurface soil:

- U.S. EPA Region 9 PRGs for Residential Soil (U.S. EPA Region 9, 2000)
- U.S. EPA generic SSLs (soil screening levels) for migration to ground water (U.S. EPA, 1996a)
- U.S. EPA generic SSLs for transfers from soil to air (U.S. EPA, 1996a)
- IDEM residential default closure levels for direct contact (IDEM, 2001)
- IDEM residential default closure levels for migration to groundwater (IDEM, 2001)

If the maximum concentration of a constituent exceeds any of these criteria (and the constituent is considered to be present at concentrations greater than basewide background levels), the chemical will be selected as a COPC for soil and carried through to the quantitative risk assessment. Given the anticipated size of the background and site-specific data-sets, it is anticipated that background comparisons will be accomplished by statistical analyses that compare background and site-specific soil data sets (see Figure 1-13). The background comparisons for purposes of COPC selection will be limited to naturally occurring inorganics only. Note that the U.S. EPA and IDEM SSLs for migration to ground water and transfers from soil to air are based on default fate and transport parameters, such as the fraction of organic carbon in soil (foc). If site-specific soil information for these parameters becomes available through the investigation proposed in this QAPP, the site-specific data will be used to recalculate the SSLs using the methodology from the U.S. EPA Soil Screening Guidance (U.S. EPA, 1996a) and IDEM RISC guidance (IDEM, 2001).

Because of the different exposure scenarios for potential human receptors, COPCs will be identified for surface and subsurface soil. Surface soil will be defined as soil collected from 0 to 2 feet below ground surface (bgs). Subsurface soil will be defined as soil collected from depths greater than 2 feet bgs.

The comparison of site soil data to U.S. EPA generic SSLs for transfers from soil to air will be used to identify whether a quantitative analysis of the inhalation of particulates or vapors from the soil exposure pathway is warranted. If the maximum soil concentration of a chemical exceeds the SSL, a quantitative evaluation of potential risks from inhalation will be performed, as described in Section C.2.4. Otherwise, the risks associated with the inhalation pathway will be considered insignificant, and the exposure pathway will be eliminated from further evaluation.

No specific screening levels exist for human exposure to sediment. COPCs will be selected for sediment by comparing detected site concentrations to the following:

- U.S. EPA Region 9 PRGs for residential soil (U.S. EPA Region 9, 2000)
- IDEM residential default closure levels for direct contact (IDEM, 2001)

A chemical detected in sediments will be selected as a COPC for sediments if the maximum detected concentration exceeds these screening values and upstream or background concentrations (see Figure 1-12). The background comparisons for purposes of COPC selection will be limited to naturally occurring inorganics only. U.S. EPA generic SSLs for transfers from soil to air and for migration to ground water are not considered to be appropriate for sediment screening because of the high moisture content

associated with sediment matrices. The use of soil screening levels for sediment COPC identification is regarded as a conservative approach because exposure to sediment is anticipated to be less than exposure to soil.

Ground Water and Surface Water

The same screening levels will be used to select COPCs for ground water and surface water. The following criteria will be used:

- U.S. EPA Region 9 PRGs for tap water (U.S. EPA, Region 9, 2000)
- IDEM residential default closure levels for groundwater (IDEM, 2001)
- U.S. EPA Maximum Contaminant Levels (MCLs) (U.S. EPA, 2000c)

If the maximum concentration of a constituent exceeds any of these criteria (and the constituent is considered to be present at concentrations greater than site-specific upgradient, upstream, or background levels), the chemical will be selected as a COPC and carried through to the quantitative risk assessment. Depending on the size of the background and site-specific ground water or surface water data sets, background comparisons will be accomplished either by a comparison of maximum site concentrations to maximum background concentrations or by statistical analyses that compare background and site-specific data sets (see Figures 1-12 and 1-14). The background comparisons for purposes of COPC selection will be limited to naturally occurring inorganics only.

Risk-based COPC screening levels for tap water ingestion, which are based on daily, residential exposure assumptions, will be used to select COPCs for ground water and surface water. In general, the use of tap water screening levels is regarded as a highly conservative approach to COPC selection at SWMUs 12, 13, 16, and 19 because ground water at the sites is not used as a potable drinking water source. Potential human exposure to the surface water in intermittent ditches and streams located within the SWMUs is expected to be limited to incidental exposures such as that which occurs during trespassing, which is significantly less than the daily exposure assumed for the ground water screening criteria.

Federal Ambient Water Quality Criteria (AWQC) will not be used to select COPCs for surface water. AWQC applicable to the protection of human health assuming routine consumption of water will not be used because the conservative U.S. EPA Region 9 PRGs for tap water, the IDEM residential closure levels for residential ground water, and the U.S. EPA MCLs will be used to select COPCs for ground

water and surface water. In addition, the surface water bodies present within and downgradient of the sites do not support game fish populations because of their size and intermittent nature.

C.1.2.2 Lead as a COPC

Limited criteria are available to evaluate the potential risks associated with lead. There are no risk-based concentrations for this chemical since the U.S. EPA has not derived toxicity values for lead. However, recommended screening levels are available for lead in soil that are used to indicate the need for response activities.

Guidance from both the Office of Prevention, Pesticides, and Toxic Substances (OPPTS) and the Office of Solid Waste and Emergency Response (OSWER) recommend 400 mg/kg as the lowest screening level for lead-contaminated soil in a residential setting, where children are frequently present (U.S. EPA, 1994a). OPPTS identifies 2,000 to 5,000 mg/kg as an appropriate range for areas where contact with soil by children in a residential setting is less frequent. A value of 400 mg/kg will be used as the screening level for soil and sediment.

Guidance from the U.S. EPA Technical Review Workgroup for Lead (TRW) indicates that "a reasonable screening level for soil lead at commercial/industrial (i.e., non-residential) sites is 750 mg/kg" for a typical non-contact intensive worker (U.S. EPA, 1999a/b). This value will not be used for COPC selection but may be used in the qualitative evaluation of lead.

The Safe Drinking Water Act Action Level of 15 µg/L (micrograms per liter) will used as the screening level for lead in ground water and surface water.

C.1.2.3 Essential Nutrients and Chemicals without Toxicity Criteria

The essential nutrients calcium, magnesium, potassium, and sodium will not be identified as COPCs because these inorganic chemicals are naturally abundant in environmental matrices and are only toxic at high doses. In addition, because of the lack of toxicity criteria, risk-based COPC screening levels are not available for some chemicals [e.g., benzo(g,h,i)perylene, phenanthrene]. These chemicals will not be selected as COPCs as they cannot be addressed during the quantitative risk assessment. However, if appropriate surrogate chemicals (which have toxicity criteria) are identified, they may be used for screening purposes, as approved. If appropriate surrogates cannot be found, the chemicals will be discussed in the data evaluation section and qualitatively addressed in the uncertainty section of the risk assessment.

C.1.2.4 Determination of Site-Related Chemicals

Chemicals found at concentrations indicative of background levels are not considered to be site-related contaminants and will not be retained as COPCs for the quantitative risk assessment. The use of basewide background soil data and site-specific background ground water, surface water, and sediment data will determine whether detected chemicals are present at naturally occurring or non-site-related anthropogenic levels. To the extent possible, conventional statistical methods (e.g., Bartlett's T-test, Wilcoxin Rank Sum, etc.) will be employed to compare site concentrations to background concentrations. If conventional statistical methods are not applicable to limited sample size or other constraints, the background comparison will be conducted as a simple comparison of maximum site concentration to maximum background concentration. Note that the background data set used for these comparisons will be the soil type from the NSWC Crane Basewide Background Soil Investigation that most closely matches the site soil samples in terms of depositional environment, depth, and grain size. If multiple soil types exist at a site, the appropriate corresponding soil type from the background data set will be used in the comparison with each site soil type.

The elimination of detected chemicals based on background data for soil will be limited to inorganics only. Although some organic compounds [primarily PAHs (polycyclic aromatic hydrocarbons), pesticides, dioxins, and PCBs (polychlorinated biphenyls)] are found at low-level concentrations in background samples, the detected concentrations are most likely reflective of non-site-related, anthropogenic sources of contamination (e.g., automobile exhausts). All detected organic compounds will be regarded as site-related for purposes of COPC selection. However, historical information for a site, the results of the analysis of soil samples collected in the reference area discussed in Section 1, and the results of the analysis of background samples will be reviewed in the baseline risk assessment to determine if the organics present in the site samples are attributable to site-related activities or anthropogenic sources.

C.1.3 COPC Summary Screening Tables

Medium-specific tables summarizing the selection of COPCs will be included in the risk assessment. The tables will be prepared according to the guidelines established for preparation of Standard Table 2 of the RAGS Part D guidance. An example format of a typical COPC selection table is provided as Table C-1.

C.2 EXPOSURE ASSESSMENT

This portion of the risk assessment defines and evaluates, quantitatively or qualitatively, the type and magnitude of human exposure to the chemicals present at or migrating from a site. The exposure

assessment is designed to depict the physical setting of the site, identify potentially exposed populations and applicable exposure pathways, calculate concentrations of COPCs to which receptors might be exposed, and estimate chemical intakes under the identified exposure scenarios.

Actual or potential exposures at SWMUs 12, 13, 16, and 19 will be determined based on the most likely pathways of contaminant release and transport, as well as human activity patterns. A complete exposure pathway has three components: a source of chemicals that can be released to the environment, a route of contaminant transport through an environmental medium, and an exposure or contact point for a human receptor.

C.2.1 Conceptual Site Model

The development of a conceptual site model (CSM) is an essential component of the exposure assessment. The CSM will integrate information regarding the physical characteristics of the site, exposed populations, sources of contamination, and contaminant mobility (fate and transport) to identify potential exposure routes and receptors to be evaluated in the risk assessment. A well-defined CSM will allow for a better understanding of the risks at a site and will aid the risk managers in the identification of the potential need for remediation. The site-specific CSMs for SWMUs 12, 13, 16, and 19 are presented in this section and illustrated in Figures C-1, C-2, C-3, and C-4. Sources of contamination, contaminant release mechanisms, transport and migration pathways, exposure routes, and potential receptors are defined. The CSMs will be refined during the risk assessment process using the data and information collected as part of the proposed field investigations. Table C-2 provides a site-specific summary of the potential receptors to be evaluated for SWMUs 12, 13, 16, and 19. A summary of the exposure routes that will be addressed quantitatively for each human receptor is also provided in Table C-2.

Site Sources of Environmental Contamination

Based on historical site data and sampling, the following parameters are among the site-related chemical contaminants known to be present or potentially present in environmental media in SWMUs 12, 13, 16, and 19:

SWMU 12

- Explosives [e.g., 2,4,6-trinitrotoluene (TNT) and cyclotetramethylene tetranitramine (HMX)] and their degradation products (e.g., 2-amino-4,6-dinitrotoluene).
- Metals (e.g., aluminum).

SWMU 13

- Explosives (e.g., TNT and HMX) and their degradation products (e.g., 2-amino-4,6-dinitrotoluene)
- Metals (e.g., aluminum)
- PCBs (from PCB oils leaked from therminol boilers)

SWMU 16

- Explosives (e.g., TNT and HMX) and their degradation products (e.g., 2-amino-4,6-dinitrotoluene)
- Metals (e.g., lead)
- PCBs (from PCB-contaminated fuels or materials)
- Petroleum hydrocarbons (from waste oils and fuels)

SWMU 19

- Explosives (e.g., 2,4,6-trinitrotoluene [TNT] and HMX) and their degradation products (e.g., 2-amino-4,6-dinitrotoluene)
- Dyes used in the testing of signaling devices
- Metals (e.g., lead)
- PCBs (from PCB-contaminated fuels or materials)
- Petroleum Hydrocarbons (from waste oils and fuels)

Potential Contaminant Release Mechanisms and Transport/Migration Pathways

Based on available historical information and a review of the existing soil data for SWMUs 12, 13, 16, and 19, a release of potentially hazardous constituents to environmental media has occurred as a result of historical site operations at these sites. For example, RDX and TNT concentrations in the surface soils of SWMUs 12, 13, and 16 exceeded residential and industrial soil clean-up levels. Because sampling and analysis of ground water at SWMU 12 and SWMU 13 have not occurred in the past, little information concerning the ground water quality and hydrogeology of these sites is available. Four monitoring wells were installed at SWMU 16 in 1983, and the ground water samples collected from these wells indicated that concentrations of several metals and trichloroethylene (TCE) exceeded drinking water criteria. Monitoring wells installed in the vicinity of Building 146 also indicate that the depth to ground water ranges from 15 to 38 feet and flows toward the south.

Given that surface and subsurface soil contamination has occurred as a result of activities at SWMUs 12, 13, 16, and 19 and that contaminants may have migrated to ground water, plausible contaminant release and migration mechanisms include the following:

SWMU 12

Media of concern at Mine Fill A have historically included air, surface water, and ground water (Halliburton NUS, 1992). Air was considered as migration pathway because particulates had been released from building ventilation systems. However, air is no longer considered a major pathway because a particulate abatement system was installed in 1973. Surface water was considered a migration pathway because explosive powders and particulates were washed off roof tops and from the railroad line near box houses, shaker rooms, and magazines. The railroad lines were hosed down once a day and, prior to the particulate abatement system, roofs were hosed off once a week. Before the installation of the carbon treatment system, wastewater from Building 153 was discharged to a settling pond from which contaminants may have eventually migrated to ground water. The wastewater also flowed from the pond to the north drain, potentially contaminating nearby surface waters. Based on the historical information, plausible contaminant release and migration mechanisms for SWMU 12 include the following:

- Transport of surface soil contaminants to the subsurface soils and ground water (and from one aquifer to another) via infiltration, percolation, and migration within the ground water aquifer.

- Overland runoff of surface waters, sediments, and surface soil from SWMU 12 via ditches and culverts to intermittent drainageways on the eastern and western borders of the Mine Fill. The intermittent drainage flows into larger intermittent drainageways that flow south-southeastward into Turkey Creek.

- Migration of contaminants in ground water (i.e., lateral migration) to potential receptor locations downgradient of SWMU 12 source areas and beyond the NSWC Crane boundary.

- Migration of fugitive dusts and VOCs from surface soils (and subsurface soils if construction and excavation activities occur).

SWMU 13

Media of concern at Mine Fill B have historically included air, surface water, and ground water (Halliburton NUS, 1992). Air was considered a migration pathway because particulates had been released from building ventilation systems. However, air is no longer considered a major pathway because a particulate abatement system was installed in 1973. Surface water was considered a migration pathway because explosive powders and particulates were washed from rooftops onto nearby soil and drainageways. In addition, explosive-contaminated wastewater was collected in sumps and released to surface drainage that mainly drains to the northwest. This drainage enters an intermittent flowing tributary of Boggs Creek, which flows in a general southward direction and exits the southern boundary of Naval Surface Warfare Center Division (NAVSURFWARCENDIV), discharging to the East Fork of the White River. Surface drainage also flows southeast of Mine Fill B and enters intermittent drainageways that flow into Turkey Creek (located approximately 1 mile from Mine Fill B). Turkey Creek flows south-southeastward and merges with Boggs Creek. It is also known that PCB-containing oils leaked from therminol boilers (removed in 1989) located near Buildings 166 and 171, contaminating the surrounding soil. Based on the historical information, plausible contaminant release and migration mechanisms for SWMU 13 include the following:

- Transport of surface soil contaminants to the subsurface soils and ground water (and from one aquifer to another) via infiltration, percolation, and migration within the ground water aquifer.

- Overland runoff of surface waters, sediments, and surface soil from SWMU 13 via ditches and culverts to intermittent drainageways on the northwestern and southeastern borders of the Mine Fill, eventually flowing into Turkey Creek and Boggs Creek.

- Migration of contaminants in ground water (i.e., lateral migration) to potential receptor locations downgradient of SWMU 13 source areas and beyond the NSWC Crane boundary.

- Migration of fugitive dusts and VOCs from surface soils and subsurface soils (if construction and excavation activities occur).

SWMU 16

Media of concern at Incinerator Building 146 have historically included air, surface water, and ground water (Halliburton NUS, 1993). The sources of contamination at SWMU 16 were the washdown and wastewater discharged through floor drains to sumps that emptied into the Boggs Creek watershed,

particulate and gaseous emissions from the deactivation furnaces prior to baghouse installation, and incinerator ash stored in piles near Building 146. Air was considered as migration pathway because particulates had been released from furnaces and ash piles. However, air is no longer considered a major pathway because the furnaces and ash piles have been removed. Surface water was considered a migration pathway because explosive powders and particulates were discharged from the floor drains of Building 146 and may have eventually entered Boggs Creek. Based on the historical information, plausible contaminant release and migration mechanisms for SWMU 16 include the following:

- Transport of surface soil contaminants to the subsurface soils and ground water (and from one aquifer to another) via infiltration, percolation, and migration within the ground water aquifer.

- Overland runoff of surface waters, sediments, surface soil, and ash via drainageways to the Boggs Creek watershed.

- Migration of contaminants in ground water (i.e., lateral migration) to potential receptor locations downgradient of SWMU 16 source areas and beyond the NSWC Crane boundary.

- Migration of fugitive dusts and VOCs from surface soils (and subsurface soils if construction/excavation activities occur).

SWMU 19

Media of concern at the Pyrotechnic Test Areas include air, surface water, soil, and ground water (Halliburton NUS, 1992). Because of the various activities that take place in the test areas, there is a complex interrelationship between waste sources, pathways, and receptors in the study areas. Smokes and contaminants are released into the atmosphere during testing. These releases could be inhaled by nearby receptors. The Rocket Range is about 2,500 feet from the southern boundary of NAVSURFWARCENDIV, and other pyrotechnic areas are about 3 miles from the facility boundaries. Boggs Creek flows through the center of the ordinance test area (OTA) and along the western edge of the Rocket Range. Both the OTA and Rocket Range are in a 100-year floodplain and are prone to flooding. Particulates released during testing could be washed into Boggs Creek during rain events. In addition, contaminated surface water could flow into the creek via drainageways on the site. Pyrotechnic materials could also leach from contaminated soil to ground water. The contaminated ground water could enter a surface water system or could migrate downward, impacting a deeper aquifer. The ground water could also serve as a drinking water source (neither facilities nor ground water supply wells exist at SWMU19 at

this time). Based on the historical information, plausible contaminant release and migration mechanisms for SWMU 19 include the following:

- Transport of surface soil contaminants to the subsurface soils and ground water (and from one aquifer to another) via infiltration, percolation, and migration within the ground water aquifer.

- Overland runoff of surface waters, sediments, surface soil via drainageways to Boggs Creek.

- Migration of contaminants in ground water (i.e., lateral migration) to potential receptor locations downgradient of SWMU 19 source areas and beyond the NSWC Crane boundary.

- Migration of fugitive dusts and VOCs during testing and from surface soils (and subsurface soils if construction and excavation activities occur).

Potential Current and Future Receptors of Concern and Exposure Pathways

NSWC Crane is an active naval base and will remain active for the foreseeable future. SWMUs 12, 13, 16, and 19 are active facilities, and operations in these facilities are expected to continue in the foreseeable future. However, for purposes of completeness, the baseline risk assessment will consider receptor exposure under residential, industrial, and recreational land use scenarios. Based on current and potential future land use, the following potential receptors may be exposed to contaminated environmental media within the study area:

- Trespassers – A plausible receptor under current or future land use. Although access to the base is controlled, once inside the base, access to the study areas is not limited by any physical constraints. In addition, hunting activities are permitted at the base. Because the study areas are relatively remote and surrounded by forested areas, hunters (particularly adolescents) may trespass within the study area. This receptor may be exposed to potentially contaminated surface soil (0 to 2 feet bgs) (incidental ingestion; dermal contact), air (inhalation), surface water (incidental ingestion; dermal contact), and sediments (incidental ingestion; dermal contact) in the intermittent streams. However, exposure to surface water is likely to be limited in some areas because of the intermittent nature of the surface water in swales and drainage ditches. Direct contact with ground water or subsurface soils is not anticipated for this receptor.

- Maintenance Workers – A plausible receptor under current and future land use. This includes adult military or civilian personnel assigned duties on a infrequent basis within the study areas (e.g., groundskeeping activities, storm sewer and drainage maintenance). This receptor could be exposed to surface soils (incidental ingestion; dermal contact), surface water (dermal contact), sediments (incidental ingestion; dermal contact), and air (inhalation). Direct contact with ground water or subsurface soils is not anticipated for this receptor.

- Construction Workers – A plausible receptor under future land use. No construction activities are currently planned for the study area. Additionally, the shallow depth to ground water in some sections of the study areas would likely preclude excavation and construction. However, excavation and construction are plausible in some sections of the study areas. Consequently, this receptor could be exposed to surface and subsurface soils (to an estimated maximum depth of 10 feet bgs) (incidental ingestion; dermal contact), ground water (dermal contact), and air (inhalation). Routine exposure to surface water and sediments is not expected for the construction worker.

- Occupational Worker – A plausible receptor under current and future land use. This includes adult military or civilian personnel assigned to routine daily work tasks. This receptor could be exposed to surface soil (incidental ingestion; dermal contact) and air (inhalation). It is anticipated that this receptor would not be routinely exposed to subsurface soils, surface waters, or sediments. Conservatively, it will be assumed that the occupational worker may be exposed to ground water (ingestion; dermal contact). (It should be noted that a public water supply is not currently available within the study area.) This receptor is expected to be exposed on a more frequent basis than the maintenance or construction worker.

- Recreational Users – A plausible receptor under future land use. If NSWC Crane were to close, the most likely scenario is that the property would be converted to a park. A recreational user may be exposed to potentially contaminated surface soil (0 to 2 feet bgs) (incidental ingestion; dermal contact), air (inhalation), surface water (incidental ingestion; dermal contact), and sediments (incidental ingestion; dermal contact). Conservatively, it will be assumed that the recreational user may be exposed to ground water (ingestion; dermal contact). (A public water supply is not currently available within the study area.) Direct contact with subsurface soils is not anticipated for this receptor. NSWC Crane is not expected to close because principal Base operations, the demilitarization of munitions, are critical to the support of the U.S. Naval fleet.

- On-Base Residents – An unlikely receptor under future land use. Although this scenario is highly unlikely, a future residential scenario is typically evaluated in a risk assessment for decision making purposes. For example, the need for deed restrictions at a site may be eliminated prior to site closure if minimal risks are estimated for residential receptors. It is assumed that a hypothetical resident may be exposed to surface soils (incidental ingestion; dermal contract), ground water (ingestion, dermal contact), surface water (ingestion; dermal contact), air (inhalation), and sediment (incidental ingestion; dermal contact).

Details regarding the assumed receptor characteristics (e.g., intake rate, frequency, duration of exposure) are defined in Section C.2.4.

C.2.2 Central Tendency Exposure vs. Reasonable Maximum Exposure

Traditionally, exposures evaluated in the human health risk assessment were based on the concept of a reasonable maximum exposure (RME) only, which is defined as "the maximum exposure that is reasonably expected to occur at a site" (U.S. EPA, December 1989). However, recent risk assessment guidance (U.S. EPA, 1992b) indicates the need to address an average case or central tendency exposure (CTE).

To provide a full characterization of potential exposure, both RME and CTE will be evaluated in the site-specific risk assessment for SWMUs 12, 13, 16, and 19. The available guidance (U.S. EPA, 1993a) concerning the evaluation of CTE is limited and at times vague. Therefore, professional judgment may be exercised when defining CTE conditions for a particular receptor at a site.

C.2.3 Exposure Point Concentrations

The exposure point concentration (EPC), which is calculated for COPCs only, is an estimate of the chemical concentration within an exposure unit that is likely to be contacted over time by a receptor and is used to estimate exposure intakes. The 95 percent upper confidence limit (UCL), which is based on the distribution of a data set, is often considered to be the best estimate of the EPC for data sets with 10 or more samples (U.S. EPA, 1992c). The 95 percent UCL will be used as the exposure concentration to assess RME and CTE risks (U.S. EPA, 1993a). For data sets with fewer than 10 samples, the UCL is considered to be a poor estimate of the mean, and the EPC is usually defined using an alternative descriptive statistic (e.g., the maximum detected concentration).

Conventional statistical methods (e.g., the Shapiro-Wilk W-Test) will be used to determine the distribution and UCL of a particular data set (Gilbert, 1987; U.S. EPA, 1992c). Detailed sample calculations, as well as general methodology for the statistical evaluation, will be presented in the site-specific risk assessment. Sample and duplicate analytical results will be averaged for statistical use. Nondetected data points will be utilized; in general, one-half the sample-specific detection limit will be employed for these analytical results.

The following paragraphs discuss the exposure units (EUs) to be evaluated and the guidelines used to calculate the medium-specific EPC.

SWMUs 12 and 13 are approximately 70 acres in area and SWMU 16 is approximately 4 acres. Given the current and anticipated land use for the study areas (i.e., military and industrial) and the rural nature of the area surrounding the base (i.e., farmland), a 2-acre EU area is considered a reasonable size. The number of EUs may be substantially reduced subject to evaluation of historical data and data obtained from this investigation. For example, SWMU 12 could contain as few as 35 EUs (based on an EU size of 2 acres). However, the majority of soil contamination at this site appears to be located around several buildings where washdown occurred (Buildings 152 and 154, for example) and the soils in the areas of the site between and beyond the building areas are assumed be "clean (i.e., concentrations are less than residential and/or industrial criteria) based on samples collected to determine the extent of contamination around the buildings at the site [Morrison Knudson (MK) Corporation, 2000]. Therefore, the number of EUs may be limited to the building areas and it may be necessary to evaluate a small number of EUs at Mine Fill A or Mine Fill B. Additional EUs may be defined based on the first soil sampling event. For example, an EU may be subdivided to gain perspective on risk estimates if significant contaminant "hot spots" are found to exist within the EU. Given that SWMU 16 is approximately 4 acres, it could be subdivided into two exposure units or more depending on the data collected in this investigation.

SWMU 19 is composed of a number of testing areas such as the cook-off areas, the firing sites, the MAPI test area, the Annex, etc. The EUs at SWMU 19 will be determined according to size and the proximity and similarity of operations. For example, the areas of the fast and slow cook-off ponds would be considered as one EU, the Super MAPI test area would be another EU, the area around Firing Site A would be another EU, etc. Using this approach, the OTA would consist of approximately 10 EUs. The Rocket Range and the Annex would represent additional separate EUs.

As detailed below, the EPC for a receptor hypothetically using or otherwise exposed to ground water underlying SWMUs 12, 13, 16, and 19 will be the arithmetic average of wells in the most highly concentrated area of the plume potentially underlying the study areas. If it is determined that a ground

water contaminant plume is or may be moving beyond the study area boundaries, EPCs for ground water at receptor locations beyond the study area may be determined via actual ground water monitoring data for wells at or near the facility boundary or by using modeling techniques. The locations will be selected, if necessary, based on the concentrations detected within study areas (i.e., the observed or potential contaminant loading to or within the ground water aquifer), the aquifer characteristics (e.g., flow, direction), and the chemical and physical nature of contaminants detected in the ground water.

The following guidelines will be used to calculate the EPCs:

- If a soil, surface water, or sediment data set for an EU contains fewer than 10 samples, the EPC for the RME and CTE case will be defined as the maximum detected concentration.

- If a soil, surface water, or sediment data set for an EU contains 10 or more samples, the 95 percent upper confidence limit (UCL) on the arithmetic mean, which will be based on the distribution of the data set, will be selected as the EPC for the RME and CTE case. Conventional statistical methods (e.g., the Shapiro-Wilk W-Test, the *t*- and *H*-statistic based UCL calculation) will be used to determine the distribution and UCL. The "best fit" distribution (normal or lognormal) will be assumed if the data set distribution is undefined. However, the EPCs calculated assuming a lognormal distribution will be reviewed and re-calculated (if necessary) as recommended in a recent EPA reference to assure that the *H*-statistic based UCL is not an over-prediction of the EPC (U.S. EPA, 1997b). If the calculated 95 percent UCL exceeds the maximum detected concentration, the maximum concentration will be used as the EPC. If enough data are available and a qualified statistician judges bootstrapping to present a more realistic estimation of risk, the bootstrapping technique described in the U.S. EPA 1997 reference will be used.

- The EPC for a ground water receptor will be the arithmetic average of wells in the highly concentrated area of the plume.

Sample and duplicate analytical results will be averaged for statistical use. Data values less than sample-specific detection limits will be substituted with one-half the detection limit. It should be noted that EPCs for ground water may also be developed for specific receptor locations (e.g., the facility boundary), as necessary, using actual ground water modeling data or ground water modeling techniques.

C.2.4 Chemical Intake Estimation

The methodologies and techniques used to estimate exposure intakes are presented in this section of the QAPP. Intakes for the identified potential receptor groups will be calculated using current U.S. EPA risk assessment guidance (e.g., U.S. EPA, 1998b, 1998a, and 1991b) and presented in the risk assessment spreadsheets. Risk assessment spreadsheets will be appended to the site-specific risk assessments as support documentation. All quantitative risk assessment results will be presented in RAGS Part D format tables.

Noncarcinogenic intakes will be estimated using the concept of an average annual exposure. Carcinogenic intakes will be calculated as an incremental lifetime exposure, which will assume a life expectancy of 70 years. Assumptions regarding exposure are presented in Table C-3.

C.2.4.1 Inhalation of Air and Fugitive Dust/Volatile Emissions from Soils

The amount of a chemical that a receptor takes in as a result of respiration is determined using the concentration of the contaminant in air. Intakes of both particulates and vapors and gases from soil will be calculated using the same equation, as follows (U.S. EPA, 1989b):

$$\text{(chemical)} \quad Intake_{ai} = (C_{ai})(IR_a)(ET)(EF)(ED)\,/(BW)(AT)$$

where:

$Intake_{ai}$	=	intake of chemical "i" from air via inhalation (mg/kg/day,)
C_{ai}	=	concentration of chemical "i" in air (mg/m^3)
IR_a	=	inhalation rate (m^3/hr)
ET	=	exposure time (hours/day)
EF	=	exposure frequency (days/yr)
ED	=	exposure duration (yr)
BW	=	body weight (kg)
AT	=	averaging time (days);
		for noncarcinogens, AT = ED x 365 days/yr;
		for carcinogens, AT = 70 yrs x 365 days/yr

The concentration of a chemical in air will be developed using modeling techniques, measured soil concentrations, and additional site-specific information.

As mentioned previously in Section C.1.2.1, a qualitative evaluation of exposure (i.e., comparison of maximum site soil concentrations to U.S. EPA generic SSLs for transfers from soil to air) will be used to identify whether a quantitative analysis of this exposure pathway is warranted. If it is determined that a quantitative evaluation is not required, the potential risks associated with the inhalation pathway will be regarded as minimal and no further evaluation will be performed.

C.2.4.2 Dermal Contact with Soil/Sediment

Direct physical contact with soil (and sediment) may result in the dermal absorption of chemicals. Exposures associated with the dermal route are estimated in the following manner (U.S. EPA 2000; U.S. EPA, 1998b; 1992a):

$$Intake_{Si} = (C_{Si})(SA)(AF)(ABS)(CF)(EF)(ED) / (BW)(AT)$$

where:

$Intake_{si}$	=	amount of chemical "i" absorbed during contact with soil/sediment (mg/kg/day)
C_{si}	=	concentration of chemical "i" in soil/sediment (mg/kg)
SA	=	skin surface area available for contact (cm^2/day)
AF	=	skin adherence factor (mg/cm^2)
ABS	=	absorption factor (dimensionless)
CF	=	conversion factor (1E-6 kg/mg)
EF	=	exposure frequency (days/yr)
ED	=	exposure duration (yr)
BW	=	body weight (kg)
AT	=	averaging time (days); for noncarcinogens, AT = ED x 365 days/yr; for carcinogens, AT = 70 yrs x 365 days/yr

Exposed surface areas of body available for dermal contact are determined on a receptor-specific basis since they correspond with assumed human activities and clothing worn during exposure events. Current guidance (U.S. EPA, 2000; U.S. EPA, 1997a; U.S. EPA, 1998b) was used to develop the default assumptions concerning the amount of skin surface area available for contact for a receptor. To maintain consistency from project to project, input parameters previously used for other NSWC Crane risk assessments (e.g., B&R Environmental, 1997) were also reviewed when developing the exposed surface areas. The rationales used to select the skin areas are as follows:

- For maintenance and occupational workers exposed to surface soil, the surface area available for soil contact is assumed to be the hands and forearms of an adult male. The skin surface area is 2,300 cm^2 for the CTE and 2,830 cm^2 for the RME. These values represent the 50th and 95th percentile areas for the hands and forearms, respectively (U.S. EPA, 1997a). The proposed adult skin surface areas for the RME and CTE case are similar to those proposed in RAGS Part E (U.S. EPA, 2000) assuming an industrial land use (3,300 cm^2 for both the RME and CTE case). The proposed adult skin surface areas are selected because the values are those presented in planning documents for other recent NSWC Crane projects (TtNUS, October 1999; TtNUS, July 2000) and because the RAGS Part E has not been finalized at this time.

- For construction workers exposed to surface and subsurface soil, the surface areas for the RME (5,800 cm^2) and CTE (5,000 cm^2) are the values recommended for soil contact by the U.S. EPA in the Exposure Factors Handbook (U.S. EPA, 1997a). These values represent 25 percent of the total body surface area of an adult male.

- For adolescent trespassers, 25 percent of the total body surface area for an adolescent (aged 6 to 16) will be assumed to be available for surface soil and/or sediment contact. The RME value (3,820 cm^2) is derived from the 95th percentile surface area data and the CTE value (3,100 cm^2) is derived from the 50th percentile data, as provided in Table 6-6 of the Exposure Factors Handbook (U.S. EPA, 1997a).

- For adult recreational users assumed to be exposed to surface soil and sediment, the exposed surface area available for contact will be the sum of the feet, lower legs, hands, and arms of an adult male. This skin surface area is 7,770 cm^2 for the CTE and 9,190 cm^2 for the RME. These values represent the 50th and 95th percentile areas for the feet, lower legs, hands, and forearms, respectively (U.S. EPA, 1997a). For a small child recreational user (0 to 6 years old), it will be assumed that 50 percent of the body surface area will be exposed to surface soil and sediment (i.e., 4,000 and 3500 cm^2, respectively) (These values represent the 95th and 50th percentile areas presented in Table 6-6 of the Exposure Factors Handbook) (U.S. EPA, 1997a).

- For adult residents exposed to surface soil and sediment, the exposed surface areas available for contact will be the U.S.-EPA-recommended values for adult skin surface area for exposure to soil (RME = 5,800 cm^2 and CTE = 5,000 cm^2 (Table 6-14, U.S. EPA, 1997a). For child residents assumed to be exposed to surface soil and sediment, the CTE and RME areas will represent 25

percent of the 50th and 95th percentile total surface area of children ages 2 to 6, respectively, as provided in Table 6-6 of the Exposure Factors Handbook (U.S. EPA, 1997a). The proposed adult skin surface areas for the RME and CTE case are similar to those proposed in RAGS Part E (U.S. EPA, 2000) assuming a residential land use (5,700 cm^2 for both the RME and CTE case). However, the skin surface areas proposed for the small child (RME = 2,000 cm^2 and CTE = 1,745 cm^2) (Table 6-6, U.S. EPA, 1997a) are less than those proposed in RAGS Part E assuming residential land use (i.e., 2,800 for both the RME and CTE case). The skin surface areas selected are those values presented in planning documents for other recent NSWC Crane projects (TtNUS, October 1999; TtNUS, July 2000) and because the RAGS Part E has not been finalized at this time.

The published ranges for the recommended soil adherence factors for the typical residential and industrial land use scenarios in RAGS Part E (U.S. EPA, 2000) are as follows:

	Central Tendency Case		**Reasonable Maximum Case**	
	Residential (mg/cm^2)	**Industrial (mg/cm^2)**	**Residential (mg/cm^2)**	**Industrial (mg/cm^2)**
Adult Receptor	0.01	0.02	0.07	0.2
Child Receptor	0.04	NA	0.2	NA

NA – Not applicable.

The adherence factors suggested for the residential land use scenario will be used to evaluate recreational and trespass exposure scenarios. Adherence factors suggested for the industrial land use scenario will be used to evaluate all types of workers considered (i.e., maintenance worker, occupational worker, and construction worker).

Current U.S. EPA guidance (U.S. EPA, 2000) will be used to determine chemical-specific dermal absorption factors.

C.2.4.3 Incidental Ingestion of Soil and Sediment

Incidental ingestion of soil (and sediment) by potential receptors coincides with dermal exposure. Exposures associated with incidental ingestion are estimated in the following manner (U.S. EPA, 1989):

$$\text{(chemicals)} \quad Intake_{Si} = (C_{Si})(IR_S)(FI)(EF)(ED)(CF)\,/(BW)(AT)$$

where:

$Intake_{si}$ = intake of contaminant "i" from soil or sediment (mg/kg/day, chem) (pCi, rad)

C_{si}	=	concentration of contaminant "i" in soil or sediment (mg/kg, chem)
IR_s	=	ingestion rate (mg/day)
FI	=	fraction ingested from contaminated source (dimensionless)
EF	=	exposure frequency (days/yr)
ED	=	exposure duration (yr)
CF	=	conversion factor (1E-6 kg/mg)
BW	=	body weight (kg)
AT	=	averaging time (days);
		for noncarcinogens, AT = ED x 365 days/yr;
		for carcinogens, AT = 70 yrs x 365 days/yr

The same exposure frequencies and durations used in the estimation of dermal intakes will be used to estimate exposure via incidental ingestion. The default values of 1.0 will be used for the fraction of soil ingested from the source for the RME and CTE. Sediment ingestion rates will be the same as those assumed for soil ingestion except that, for the resident, the proposed sediment ingestion rate will be 50 percent of the proposed soil ingestion rate.

C.2.4.4 Dermal Contact with Ground Water and Surface Water

The same equation is used to estimate intakes for dermal contact with ground water and surface water. Direct contact with ground water at SWMUs 12, 13, 16, and 19 is limited to exposure that would occur under a residential, recreational, typical industrial (occupational), and construction scenarios. Hypothetical future on-base residential receptors are assumed to use ground water for domestic purposes (i.e., bathing, showering, washing dishes) that can result in a dermal exposure. It is also assumed that off-base residents may use the ground water resource for domestic purposes. Short-term dermal exposure is assumed to occur for the construction worker during excavation activities and for hypothetical recreational user or occupational worker receptors that may occasionally use restroom facilities (supplied by ground water pumped from the underlying aquifer) while working or recreating. (It should be noted that neither facilities nor ground water supply wells exist at SWMUs 12, 13, 16, and 19 at this time). Dermal contact with surface water may also occur while receptors are involved in certain activities, such as landscaping (maintenance worker), trespassing, or recreational sport (hiking, biking, etc.).

The following equation will be used to assess exposures resulting from dermal contact with water (U.S. EPA, 2000):

$$DAD_{wi} = (DA_{event})(EV)(ED)(EF)(A)/(BW)(AT)$$

where:

DAD_{wi}	=	dermally absorbed dose of chemical "i" from water (mg/kg/day)
DA_{event}	=	absorbed dose per event (mg/cm^2-event)
EV	=	event frequency (events/day)
ED	=	exposure duration (yr)
EF	=	exposure frequency (days/yr)
A	=	skin surface area available for contact (cm^2)
BW	=	body weight (kg)
AT	=	averaging time (days);
		for noncarcinogens, AT = ED x 365 days/yr;
		for carcinogens, AT = 70 yrs x 365 days/yr

Ground water exposure for hypothetical future on-base residential receptors using the ground water as a domestic water supply is assumed to occur on a daily basis. Exposure to ground water for construction workers, recreational users, or typical occupational workers and exposure to surface water for maintenance workers, trespassers, and recreational users are also assumed to occur each day the receptor visits the study area. Dermal intakes for residents will assume total body exposure. For construction workers, trespassers, occupational workers, maintenance workers, and recreational users, the exposed surface area of the body available for contact was based on assumed activities and is similar to the assumptions outlined for dermal contact with soil and sediment. Table C-3 summarizes recommendations for exposure parameters that will be evaluated in the baseline risk assessment.

The absorbed dose per event (DA_{event}) will be estimated using a non-steady-state approach for organic compounds and a traditional steady-state approach for inorganics. For organics, the following equations apply:

$$\text{If } t_{event} < t^*, \text{ then}: DA_{event} = (2K_p)FA(C_{wi})(CF)\left[\frac{\sqrt{6\,T\,t_{event}}}{\pi}\right]$$

$$\text{If } t_{event} > t^*, \text{ then}: DA_{event} = (K_p)FA(C_{wi})(CF)\left[\frac{t_{event}}{1+B} + 2T\left[\frac{1+3B+3B^2}{(1+B)^2}\right]\right]$$

where:

t_{event}	=	duration of event (hr/event)
t^*	=	time it takes to reach steady-state conditions (hr)

K_p	=	permeability coefficient from water through skin (cm/hr)
C_{wi}	=	concentration of chemical "i" in water (mg/L)
T	=	lag time (hr)
π	=	constant (dimensionless; equal to 3.1416)
CF	=	conversion factor (1E-3 L/cm^3)
B	=	Dimensionless ratio of the permeability of the stratum corneum relative to the permeability across the viable epidermis.
FA	=	Chemical specific fraction absorbed (dimensionless)

Values for the chemical-specific parameters (t_{event}, t^*, K_p, T, FA, and B) will be obtained from RAGS Part E the current dermal guidance (U.S. EPA, 2000 and updates). If no published values are available for a particular compound; they will be calculated using equations provided in the cited guidance.

The following non-steady-state equation will be used to estimate DA_{event} for inorganics:

$$DA_{event} = (K_p)\,(C_{wi})\,(t_{event})$$

In general, the recommended default value of 1E-3 will be used for the dermal permeability of inorganic constituents. For most metals, dermal absorption is not a significant pathway because penetration through the skin is minimal.

C.2.4.5 Incidental and Direct Ingestion of Ground Water and Surface Water

Potential receptors may be exposed to ground water or surface water via direct ingestion. Intakes associated with ingestion of water will be evaluated using the following equations (U.S. EPA, 1989):

$$\text{(chemical)}\quad \text{Intake}_{wi} = (C_{wi})(IR_w)(EF)(ED)/(BW)(AT)$$

where:

Intake_{wi}	=	intake of chemical "i" from water (mg/kg/day, chem)
C_{wi}	=	concentration of chemical "i" in water (mg/L, chem)
IR_w	=	ingestion rate for ground water (L/day)
IR_w	=	ingestion rate for surface water (L/day) = (CR)(ET)
CR	=	contact rate for surface water (L/hr)
ET	=	exposure time for surface water (hr/day)

EF	=	exposure frequency (days/yr)
ED	=	exposure duration (yr)
BW	=	body weight (kg)
AT	=	averaging time (days);
		for noncarcinogens, AT = ED x 365 days/yr;
		for carcinogens, AT = 70 yrs x 365 days/yr

The same exposure times, frequencies, and durations used to assess dermal exposure to water will be used to estimate intakes for ingestion of water.

C.2.4.6 Inhalation of Volatiles in Ground Water

Ground water exposure may also result in an inhalation exposure if the water resource is used as a domestic water supply. This exposure route will be evaluated for residential receptors only who may be exposed while showering, bathing, washing dishes, etc. Inhalation exposures will be estimated using a mass transfer model, developed specifically for this exposure route, in combination with an air intake estimation model. The mass transfer model accounts for inhalation that occurs during a shower and after a shower while the receptor remains in the closed bathroom. The method that is employed is as follows (U.S. EPA, 1989; Foster and Chrostowski, 1987):

$$Intake_{si} = (S)(IR_{sh})(K)(EF)(ED) / (BW)(AT)(R_a)(CF)$$

$$K = D_s + \frac{\exp(R_a \times D_t)}{R_a} - \frac{\exp R_a \times (D_s - D_t)}{R_a}$$

where:

$Intake_{wi}$	=	intake of chemical "i" from water via inhalation (mg/kg/day)
S	=	volatile chemical generation rate (µg/m^3-min-shower)
IR_{sh}	=	inhalation rate (L/min)
K	=	mass transfer coefficient (min)
EF	=	exposure frequency (showers/yr)
ED	=	exposure duration (yrs)
BW	=	body weight (kg)
AT	=	averaging time or period of exposure (days)
R_a	=	air exchange rate (min^{-1})

CF	=	conversion factor ($1x10^{-6}$ µg-L/mg-m^3)
D_s	=	shower duration (min)
D_t	=	total time in bathroom (min)

The volatile chemical generation rate will be estimated using the Foster and Chrostowski mass transfer model, which is based on two-phase film theory. The model employs contaminant-specific mass transfer coefficients, Henry's Law constants, droplet diameter, drop time, viscosity, temperature, etc. Calculations of the shower model will be provided in the appendices to the site-specific risk assessments.

C.2.4.7 Summary of Exposure Parameters

A summary of the exposure input parameters for all exposure pathways are presented in Table C-3 for the identified potential receptor groups at SWMUs 12, 13, 16, and 19. In general, standard default parameters (e.g., U.S. EPA, 1991a; Exposure Factors Handbook, U.S. EPA 1997a), which combine mid-range and upper-end exposure factors, will be used to assess RME conditions. CTE will be assessed primarily by the use of mid-range exposure factors presented in current risk assessment guidance (U.S. EPA, 1989 and 1993a). These input parameters may be updated according to new risk assessment guidance, as it becomes available.

C.2.5 Exposure to Lead

The equations and methodology presented in the previous section cannot be used to evaluate exposure to lead because of the absence of published dose-response parameters. Exposure to lead will be assessed using the following models:

- The latest version of U.S. EPA's Integrated Exposure Uptake Biokinetic (IEUBK) Model for lead, Version 1.0 (U.S. EPA, 1994a and 2001) is typically used to evaluate lead exposure assuming a residential land use scenario.

- The U.S. EPA's Technical Review Workgroup (TRW) Model for Lead (U.S. EPA, 1996c). This model is typically used to evaluate lead exposure assuming a non-residential land use scenario.

The IEUBK Model for lead (U.S. EPA, 1994a and 2001) is designed to estimate blood levels of lead in children (under 7 years of age) based on either default or site-specific input values for air, drinking water, diet, dust, and soil exposure. Studies indicate that infants and young children are extremely susceptible to adverse effects from exposure to lead. Considerable behavioral and developmental impairments have

been noted in children with elevated blood-lead levels. The threshold for toxic effects from this chemical is believed to be in the range of 10 µg/dL to 15 µg/dL. Blood-lead levels greater than 10 µg/dL are considered to be a "concern."

For SWMUs 12, 13, 16, and 19, the IEUBK Model for lead will be used to address exposure to lead in children when detected ground water and surface water concentrations exceed the 15 µg/L Federal Action Level promulgated under the Safe Drinking Water Act and when detected soil and sediment concentrations exceed the OSWER soil screening level of 400 mg/kg for residential land use (U.S. EPA, 1994b). Average chemical concentrations, as well as default parameters for some input parameters, will be employed. Estimated blood-lead levels and probability density histograms will be presented as support documentation for this analysis and appended to the site-specific risk assessment.

Non-residential adult exposure to lead in soil will be evaluated using U.S. EPA's Technical Review Workgroup for Model Lead (U.S. EPA, 1996c). In this model, adult exposure to lead in soil is addressed by an evaluation of the relationship between the site soil lead concentration and the blood-lead concentration in the developing fetuses of adult women. The adult lead model will generate a spreadsheet for each exposure scenario that is evaluated (i.e., industrial, recreational). The output of the spreadsheet will be the probability that the blood-lead concentrations in the fetus exceed 10 µg/L. The probability that the fetal blood-lead level will exceed 10 µg/L will be calculated in accordance with the following EPA guidelines:

- Use of the TRW Interim Adult Lead Methodology in Risk Assessment. Memorandum from Pat Van Leeuwen (Region 5 Superfund Program) and Paul White (ORD/NCEA) to Mark Maddaloni, Chair, TRW Adult Lead Subgroup, April 7, 1999.

- Frequently Asked Questions (FAQs) on the Adult Lead Model. Technical Review Workgroup for Lead, Guidance Document, April 1999, Rev. 0.

No models are currently available to evaluate the periodic exposure of adolescent trespassers to lead. Therefore, the results of the IEUBK Model for children will be used to qualitatively assess exposure of this receptor. Essentially, the qualitative discussion will cite that potential adverse effects from exposure to lead are expected to be of a lesser magnitude for adolescent trespassers than for children.

C.3 TOXICITY ASSESSMENT

The objective of the toxicity assessment is to identify the potential health hazards and adverse effects in exposed populations. Quantitative estimates of the relationship between the magnitude and type of exposures and the severity or probability of human health effects will be defined for the identified COPCs. Quantitative toxicity values determined during this component of the risk assessment will be integrated with outputs of the exposure assessment to characterize the potential for the occurrence of adverse health effects for each receptor group.

The toxicity value used to evaluate noncarcinogenic health effects is the reference dose (RfD). Carcinogenic effects are quantified using the cancer slope factor (CSF).

C.3.1 Toxicity Criteria

Oral and inhalation RfDs and CSFs to be used in the site-specific risk assessments for SWMUs 12, 13, 16, and 19 will be obtained from the following primary literature sources:

- Integrated Risk Information System (IRIS).
- Annual Health Effects Assessment Summary Tables (HEAST).
- National Center for Environmental Assessment (NCEA) Superfund Health Risk Technical Support Center.

Although RfDs and CSFs can be found in several toxicological sources, U.S. EPA's IRIS on-line database is the preferred source of toxicity values. This database is continuously updated and presented values have been verified by U.S. EPA RfD and Carcinogenic Risk Assessment Verification Endeavor (CRAVE) work groups. The U.S. EPA Region 9 PRG Tables and Region 3 RBC Tables will also be used as a source of toxicity criteria when toxicity criteria are not available from the aforementioned references.

C.3.1.1 Toxicity Criteria for Dermal Exposure

RfDs and CSFs found in literature may be expressed as administered doses; therefore, these values are considered to be inappropriate for estimating the risks associated with dermal routes of exposure. Oral dose-response parameters based on administered doses must be adjusted to absorbed doses before the comparison to estimated dermal exposure intakes is made.

The adjustment from administered to absorbed dose will be made using chemical-specific absorption efficiencies published in available guidance [i.e., U.S. EPA, 2000 (the primary reference), IRIS, Agency for Toxic Substances and Disease Registry (ATSDR) toxicological profiles, etc.] and the following equations:

$$RfD_{dermal} = (RfD_{oral})(ABS_{GI})$$

$$CSF_{dermal} = (CSF_{oral}) / (ABS_{GI})$$

where: ABS_{GI} = absorption efficiency in the gastrointestinal tract

Absorption efficiencies used in the risk assessments will reflect U.S. EPA's current dermal assessment guidance (U.S. EPA, 2000).

C.3.1.2 Toxicity Criteria for Carcinogenic Effects of PAHs

Limited toxicity values are available to evaluate the carcinogenic effects from exposure to PAHs. The most extensively studied PAH is benzo(a)pyrene, which is classified by U.S. EPA as a known human carcinogen. Although CSFs are available for benzo(a)pyrene, insufficient data are available to calculate CSFs for other carcinogenic PAHs. Toxic effects for these chemicals will be evaluated using the concept of estimated orders of potential potency, as presented in current U.S. EPA guidance (U.S. EPA, 1993b). These parameters are based on the carcinogenicity of benzo(a)pyrene and are available for select carcinogenic PAHs. The equivalent oral and inhalation CSF for these chemicals is derived by multiplying the CSF for benzo(a)pyrene by the order of potential potency.

C.3.1.3 Toxicity Criteria for Carcinogenic Effects of Dioxins/Furans

Similar to the concept of estimated orders of potential potency for PAHs, chlorinated dibenzo-p-dioxins (CDDs) and -dibenzofurans (CDFs) will be evaluated using Toxicity Equivalence Factors (TEFs) relative to the toxicity of 2,3,7,8-tetrachlorodibenzo-p-dioxin (TCDD) (Environmental Health Perspectives, December 1998; U.S. EPA, March 1989). Based on a variety of approaches that generate toxicities relative to 2,3,7,8-TCDD, the U.S. EPA and others have developed TEFs for other dioxins and furans from structure-activity relationships and the available toxicological information. An equivalent oral CSF for these chemicals may be derived by multiplying the CSF for 2,3,7,8-TCDD by the TEF. However, the TEFs are more typically used to derive a toxicity equivalent concentration (TEC) representing all the

significant dioxin and furans in a sample. An EPC based on the TECs for a dataset is then evaluated using the CSF for 2,3,7,8-TCDD.

C.3.1.4 Toxicity Criteria for Chromium

Toxicity criteria are available for different forms of chromium, which is considered to be more toxic in the hexavalent state. Because there is no evidence to support the conclusion that hexavalent chromium is present at the sites, speciation analyses will not be completed for SWMUs 12, 13, 16, and 19. Risks associated with this chemical will be assessed by assuming that 10 percent of the reported total chromium result is attributable to hexavalent chromium, and 90 percent of the total chromium result comprises trivalent chromium. This assumption is based on published toxicological information (ATSDR, 1991).

C.3.2 Toxicity Profiles

Toxicological profiles for each COPC will be presented as an appendix to the risk assessment. These brief profiles present a summary of the current available literature on the carcinogenic and noncarcinogenic health effects associated with human exposure to COPCs.

C.4 RISK CHARACTERIZATION

Potential risks (noncarcinogenic and carcinogenic) for human receptors resulting from the exposures outlined in the exposure assessment are quantitatively determined during the risk characterization component of the baseline human health risk assessment.

A summary and interpretive discussion of the quantitative risk estimates will be provided in the text of the site-specific risk assessments. During the interpretive risk discussion, COPCs that contribute significantly to elevated risks will be identified as "risk drivers" or chemicals of concern (COCs). All the numeric estimates of risk will be contained in the risk assessment spreadsheets, which will be appended to the assessment as support documentation.

C.4.1 Quantitative Analysis

Quantitative estimates of risk will be calculated according to risk assessment methods outlined in U.S. EPA guidance (U.S. EPA, 1989). Lifetime cancer risks will be expressed in the form of dimensionless probabilities, referred to as incremental cancer risks (ICRs), based on CSFs. Noncarcinogenic risk

estimates will be presented in the form of Hazard Quotients (HQs) that are determined through a comparison of intakes with published RfDs.

ICR estimates are generated for each COPC using estimated exposure intakes and published CSFs, as follows:

$$ICR = (\text{Estimated Exposure Intake})(CSF)$$

If the above equation results in an ICR greater than 0.01, the following equation will be used:

$$ICR = 1-[\exp(-\text{Estimated Exposure Intake})(CSF)]$$

An ICR of 1E-6 indicates that the exposed receptor has a one-in-one-million chance of developing cancer under the defined exposure scenario. Alternatively, such a risk may be interpreted as representing one additional case of cancer in an exposed population of one million persons.

As mentioned previously, noncarcinogenic risks will be assessed using the concept of HQs and Hazard Indices (HIs). The HQ for a COPC is the ratio of the estimated intake to the RfD, as follows:

$$HQ = (\text{Estimated Exposure Intake})/(RfD)$$

An HI will be generated by summing the individual HQs for all COPCs. The HI is not a mathematical prediction of the severity of toxic effects and therefore is not a true "risk"; it is simply a numerical indicator of the possibility of the occurrence of noncarcinogenic (threshold) effects.

C.4.1.1 Comparison of Quantitative Risk Estimates to Benchmarks

To interpret the quantitative risks and to aid risk managers in determining the need for remediation at a site, quantitative risk estimates will be compared to typical benchmarks. Calculated ICRs will be interpreted using the U.S. EPA's "target range" ($1x10^{-6}$ to $1x10^{-4}$), and HIs will be evaluated using a value of 1.0.

U.S. EPA has defined the range of $1x10^{-6}$ to $1x10^{-4}$ as the ICR "target range" for most hazardous waste facilities addressed under CERCLA and RCRA. Individual or cumulative ICRs greater than 1E-4 will typically not be considered as protective of human health, and ICRs less than 1E-6 will typically be

regarded as protective. (Risk management decisions are necessary when the ICR is within the 1E-4 to 1E-6 cancer risk range.)

An HI exceeding unity (1.0) indicates that there may be potential noncarcinogenic health risks associated with exposure. If an HI exceeds unity, a segregation of target organs effects associated with exposure to COPCs will be performed. Only those chemicals that affect the same target organ(s) or exhibit similar critical effect(s) will be regarded as truly additive. Consequently, it may be possible for a cumulative HI to exceed 1.0, but no adverse health effects are anticipated if the COPCs do not affect the same target organ or exhibit the same critical effect.

As a general guideline, a no-further-action recommendation will be forwarded to the Navy, the state of Indiana, and U.S. EPA if the cancer risk estimates and total HIs (developed on a target organ/target effect basis) for receptors of concern do not exceed $1x10^{-4}$ and 1, respectively. Otherwise, the need for remedial action (including institutional controls) will be evaluated in the corrective measures study (CMS). However, as indicated in EPA RAGS Part D, the upper boundary of the acceptable risk range is not a discrete line at $1x\ 10^{-4}$. "Risks slightly greater than $1x10^{-4}$ may be considered to be acceptable (i.e., protective) if justified based on site-specific conditions, including any uncertainties about the nature and extent of contamination and associated risks." Consequently, a no-further-action recommendation may be forwarded even when the $1x\ 10^{-4}$ risk benchmark is exceeded. The following factors will be considered in this determination:

- The magnitude of the medium-specific risk estimates.

- Significant uncertainties in the baseline human health risk assessment that would tend to overestimate baseline risk assessment results. Uncertainties in the baseline human health risk assessment intake estimates (and their impact on the risk estimates) could be evaluated using probabilistic risk assessment techniques. Uncertainties associated with the toxicity criteria would be evaluated qualitatively.

- Significant uncertainties in the EPC estimates that would tend to overestimate baseline risk assessment results.

C.4.2 Qualitative Analysis

As mentioned previously, a qualitative evaluation of risk will be made for several exposure situations.

- The soil inhalation pathway will be initially evaluated by a comparison of maximum site soil concentrations to U.S. EPA generic SSLs for transfers from soil to air. If the maximum site concentration exceeds the SSL for a chemical, a quantitative analysis of this exposure pathway will be performed.

- The potential for the migration of soil contaminants to ground water will be assessed by a comparison of maximum and average soil concentrations to U.S. EPA generic SSLs for transfers from soils to ground water. SSLs based on dilution and attenuation factors (DAFs) of 1 and 20 will be used in the evaluation.

C.5 UNCERTAINTY ANALYSIS

The goal of the uncertainty analysis is to identify important uncertainties and limitations associated with the baseline human health risk assessment. Uncertainties related to each component of the assessment (i.e., data evaluation, exposure assessment, toxicity assessment, and risk characterization) will be presented. In addition, the effect of a particular uncertainty on the outcome of the assessment (i.e., risk estimates) will also be indicated, where possible. The following subsections present an overview of uncertainties that may be addressed in the risk assessment uncertainty section.

C.5.1 Uncertainty in Data Evaluation

This section may discuss uncertainties in the risk assessment associated with the analytical data and data quality. This may also involve a discussion of uncertainty in the COPC selection process, the inclusion or exclusion of COPCs in the risk assessment on the basis of background concentrations, the uncertainty in COPC screening levels, and the omission of constituents for which health criteria are not available. The presented discussion will be based, in part, on the evaluation presented in the Data Useability Worksheet, as suggested by U.S. EPA RAGS Part D.

C.5.2 Uncertainty in the Exposure Assessment

This section will include a discussion of the following: assumptions related to current and future land use, the uncertainty in exposure point concentrations, (for example, the use of maximum concentrations to estimate risks) uncertainty in the selection of potential receptors and exposure scenarios, and uncertainty in the selection of exposure parameters (RME vs. CTE). If predictive models are used in the risk estimation, the uncertainty associated with the model and modeling parameters will be evaluated.

C.5.3 Uncertainty in the Toxicity Assessment

The uncertainties inherent in RfDs and CSFs and use of available criteria will be discussed. A discussion of the uncertainty in hazard assessment, which deals with characterizing the nature and strength of the evidence of causation or the likelihood that a chemical that induces adverse effects in animals will also induce adverse effects in humans, will be provided. This section will also discuss uncertainty in the dose-response evaluations for the COPCs that relates to the determination of a CSF for the carcinogenic assessment and derivation of an RfD or Reference Concentration (RfC) for the noncarcinogenic assessment. In addition, a discussion of the uncertainty in the toxicity of specific constituents, such as PAHs, arsenic, chromium, aluminum, iron, and copper, will be presented, if applicable.

C.5.4 Uncertainty in the Risk Characterization

This section will discuss the uncertainty in risk characterization that results primarily from assumptions made regarding additivity and synergism of effects from exposure to multiple COPCs affecting different target organs across various exposure routes. The risk assessment will discuss the uncertainty inherent in summing risks for several substances across different exposure pathways. It should be noted that probabilistic risk assessment techniques may also be used to further define the uncertainty attached to the risk characterization results. However, the exposure assumptions (e.g., probability distributions) used to prepare the probabilistic risk assessment will be reviewed with the regulatory reviewers before they are incorporated into the uncertainty section of the baseline risk assessment.

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TABLE C-1

OCCURRENCE, DISTRIBUTION AND SELECTION OF CHEMICALS OF POTENTIAL CONCERN
SWMUs 12/13/16
NSWS CRANE, CRANE, INDIANA

Scenario Timeframe:
Medium:
Exposure Medium:
Exposure Point:

CAS Number	Chemical	Minimum Concentration (1)	Minimum Qualifier	Maximum Concentration (1)	Maximum Qualifier	Units	Location of Maximum Concentration	Detection Frequency	Range of Detection Limits	Concentration Used for Screening	Background Value (2)	Screening Toxicity Value (3)	Potential ARAR/TBC Value	Potential ARAR/TBC Source	COPC Flag	Rationale for Contaminant Deletion or Selection (4)

1 Minimum/maximum defected concentration.

2 N/A - Refer to supporting information for background discussion.

Background values derived from statistical analysis. Follow Regional guidance and provide supporting information.

3 Provide reference for screening toxicity value.

4 Rationale Codes Selection Reason: Infrequent Detection but Associated Historically (HIST).
Frequent Detection (FD)
Toxicity Information Available (TX0
Above Screening Levels (ASL)
Deletion Reason: Infrequent Detection (IFD)
Background Levels (BKG)
No Toxicity Information (NTX)
Essential Nutrient (NUT)
Below Screening Level (BSL)

Definitions: N/A = Not Applicable
SQL = Sample Quantitation Limit
COPC = Chemical of Potential Concern
ARAC/TBC = Applicable or Relevant and Appropriate Requirement/To Be Considered
MCL = Federal Maximum Contaminant Level
SMCL = Secondary Maximum Contaminant Level
J = Estimated Value
C = Carcinogenic
N = Non-Carcinogenic

TABLE C-2

EXPOSURE ROUTES FOR QUANTITATIVE EVALUATION
SWMUs 12, 13, 16, AND 19
NSWC CRANE, CRANE INDIANA

Receptors	Exposure Routes
Adolescent Trespassers (6 to 17 Years) (current/future land use)	• Soil dermal contact (surface) • Soil ingestion (surface) • Inhalation of air/dust/emissions (surface) • Surface water/sediment dermal contact • Surface water/sediment Ingestion
Maintenance Workers (current and future land use)	• Soil dermal contact (surface) • Soil ingestion (surface) • Inhalation of air/dust/emissions (surface) • Surface water/sediment dermal contact
Construction Workers (future land use)	• Soil dermal contact (surface and subsurface) • Soil ingestion (surface and subsurface) • Inhalation of air/dust/emissions (surface and subsurface) • Ground water dermal contact (during excavation) • Ground water inhalation of volatile organics (during excavation)
Small Child (0 to 6 yrs.) and Adult Recreational Users (future land use)	• Soil dermal contact (surface) • Soil ingestion (surface) • Inhalation of air/dust/emissions (surface) • Surface water/sediment dermal contact • Surface water/sediment ingestion • Ground water dermal contact • Direct ingestion of ground water
On-Base Residents (Adult/Children) (future land use)	• Soil dermal contact (surface) • Soil ingestion (surface) • Inhalation of air/dust/emissions (surface) • Direct ingestion of ground water • Ground water dermal contact (showering/bathing) • Inhalation of volatiles in ground water (showering/bathing) • Surface water/sediment dermal contact • Surface water/sediment ingestion
Occupational Worker (current and future)	• Soil dermal contact (surface) • Soil ingestion (surface) • Inhalation of air/dust/emissions (surface) • Direct ingestion of ground water • Ground water dermal contact

TABLE C-3

SUMMARY OF EXPOSURE ASSUMPTIONS
SWMUs 12/13/16/19
NSWC CRANE, CRANE, INDIANA

Receptor	Exposure Assumptions			Rationale/Reference
	Input Parameter	RME	CTE	
Adolescent Trespasser (6 to 17 Years)	EF (days/yr)	26	13	Professional judgement; 1 day/week in warm weather months for the RME and 1 day/every other week in warm weather months for the CTE.
	ED (yrs)	11	11	Adolescents from age 6 to 17 evaluated.
	ET (hours/day) and t_{event} (hr/event)	4	2	Professional judgement.
	BW (kg)	43	43	Average age-specific value (USEPA, May 1989).
	SA (cm^2/day) and A (cm^2)1	3,820	3,100	25 percent of the total body surface area will be assumed to be available for soil, sediment, and surface water contact. CTE and RME areas represent the mean of 50th and 95th percentile values for ages 6 to 17, respectively, as provided in Table 6-6 of the Exposure Factors Handbook (USEPA, August 1997).
	CR (L/hr)	0.05	0.05	Intake rates for exposure to surface water during swimming (USEPA, November 1995).
	IR_s/IR_{sed} (mg/day)	100	50	Assumed similar to adult exposure (USEPA, May 1993).
	IR_a (m^3/hour)	1.9	1.9	Recommended short-term heavy activities rate for children (USEPA, August 1997; Table 5-23).
Maintenance/ Occupational Worker	EF (days/yr)	24 (maintenance) 250 (occupational)	12 (maintenance) 219 (occupational)	Professional judgement for maintenance worker; 2 days/month for the RME and 1 day/month for the CTE. Convention for the occupational worker (USEPA, May 1993).
	ED (yrs)	25	9	Convention for RME (USEPA, March 1991) and CTE (USEPA, May 1993).
	ET (hours)	8 (air) (occupational worker) 4 (air) (maintenance worker) 2 (surface water) (maintenance worker) 0.25 (ground water) (occupational worker)	8 (air) (occupational worker) 4 (air) (maintenance worker) 1.5 (surface water) (maintenance worker) 0.167 (ground water) (occupational worker)	Standard default for occupational worker exposed to air. (USEPA, March 1991). Professional judgement for all other parameters.
	BW (kg)	70	70	USEPA, May 1993.

TABLE C-3

SUMMARY OF EXPOSURE ASSUMPTIONS
SWMUs 12/13/16/19
NSWC CRANE, CRANE, INDIANA

Receptor	Exposure Assumptions			Rationale/Reference
	Input Parameter	RME	CTE	
Maintenance/ Occupational Worker (Continued)	SA (cm^2/day)	2,830	2,300	Surface area of the hands and forearms measured in men assumed to be available for soil/sediment/surface water contact. The RME and CTE values represent the 95th and 50th percentile areas of the hands and forearms (USEPA, August 1997, Table 6-2). (Similar to recommended SA for industrial worker Table 3.5, RAGS Part E.)
	IR_{sed}/IR_s (mg/day)	100	50	USEPA, May 1993.
	IR_a (m^3/hour)	2.5 (maintenance) 0.6 (occupational)	2.5 (maintenance) 0.6 (occupational)	For maintenance workers, recommended short-term heavy activities rate for outdoor workers (USEPA, August 1997; Table 5-23). For occupational workers, adult daily inhalation rate (USEPA, December 1989 and page 5-24 of USEPA, August 1997).
	IR_w (L/day)	1 (occupational worker)	1 (occupational worker)	Professional judgement.
Construction Worker	EF (days/yr)	150	150	Professional judgement. Ground assumed to be frozen or snow covered for 22 weeks/yr.
	ED (yrs)	1	1	Estimated length of construction project (professional judgement).
	ET (hours)	8 (air) 2 (ground water)	8 (air) 1.5 (ground water)	For air, standard default (USEPA, March 1991). Professional judgement for dermal exposure to ground water.
	BW (kg)	70	70	USEPA, May 1993.
	SA (cm^2/day)	5,800	5,000	Recommended values for adult skin surface area assumed to be available for soil contact (USEPA, August 1997; Table 6-16). Valued also assumed for dermal contact with ground water.
	IR_s (mg/day)	480	240	Convention for the RME (USEPA, March 1991). CTE is assumed to be one-half the RME value.
	IR_a (m^3/hour)	2.5	2.5	Recommended short-term heavy activities rate for outdoor workers (USEPA, August 1997; Table 5-23).
	EV (events/day)	1	1	Professional judgement.
Small Child and Adult Recreational User	EF (days/yr)	52	26	Professional judgement; 2 day/week for the RME in warm weather months and 1 day/every other week in warm weather months for the CTE.
	ED (yrs)	24(1) 6(3)	7(1) 2(3)	USEPA, May 1993. Assumed length of residence for an adult living near the facility.
	ET (hours/day) and t_{event} (hr/event)	4	2	Professional judgement.

TABLE C-3

SUMMARY OF EXPOSURE ASSUMPTIONS
SWMUs 12/13/16/19
NSWC CRANE, CRANE, INDIANA

Receptor	Exposure Assumptions			Rationale/Reference
	Input Parameter	RME	CTE	
Small Child and Adult Recreational User (Continued)	BW (kg)	70[1] 15[3]	70[1] 15[3]	USEPA, May 1993.
	SA (cm^2/day)	9,190 (adult) 4,000 (child)	7,770 (adult) 3,500 (child)	Feet, lower legs, hands, and arms of adult male assumed to be available for soil, surface water, and sediment contact. The RME and CTE values represent the 95th and 50th percentile areas of the feet, lower legs, hands and arms (USEPA, August 1997, Table 6-2). Fifty-percent of the total body surface area for child will be assumed to be available for soil, sediment, and surface water contact. CTE and RME represent the mean of the 50th and 95th for ages 0 to 6 as provided in Table 6-6 of the Exposure Factors Handbook (August 1997).
	IR_{sed}/IR_S (mg/day)	200[3] 100[1]	100[3] 50[1]	Based on USEPA, May 1993.
	IR_a (m^3/hour)	1.2[3] 1.6[1]	1.2[3] 1.6[1]	Recommended short-term moderate activities rate for adults and children (USEPA, August 1997 page 5-24 and Table 5-23).
	IR_w (l/DAY)	1	1	Professional judgment.
	CR (L/hr)	0.05[1] 0.01[2]	0.05[1] 0.01[2]	Intake rates for exposure to surface water during swimming (USEPA, November 1995).
	FI	0.5	0.5	Professional judgment.
Resident (Adult/Small Child)	EF (days/yr or showers/yr)	350	234	USEPA, May 1993. One shower assumed to be taken per day.
	ED (yrs)	24[1] 6[3]	7[1] 2[3]	USEPA, May 1993.
	t_{event} (hr/event) (showering)	0.25	0.167	15 min/event for RME and 10 min/event for CTE (USEPA, January 1992).
	BW (kg)	70[1] 15[3]	70[1] 15[3]	USEPA, May 1993.
	SA (cm^2/day)	5,800[1] [5] 2,000[3] [5] 18,000[1] [4] 6,600[1] [4]	5,000[1] [5] 1,745[3] [5] 18,000[1] [4] 6,600[1] [4]	Recommended values for adult skin surface area assumed to be available for soil or sediment contact (USEPA, August 1997). [Child CTE and RME areas represent 25% of the total body area, as provided in Table 6-6 of the Exposure Factors Handbook (USEPA, August 1997)]. Recommended values for domestic use of ground water or surface water based on Exhibit 3.2 (RAGS Part E2000).

TABLE C-3

SUMMARY OF EXPOSURE ASSUMPTIONS
SWMUs 12/13/16/19
NSWC CRANE, CRANE, INDIANA

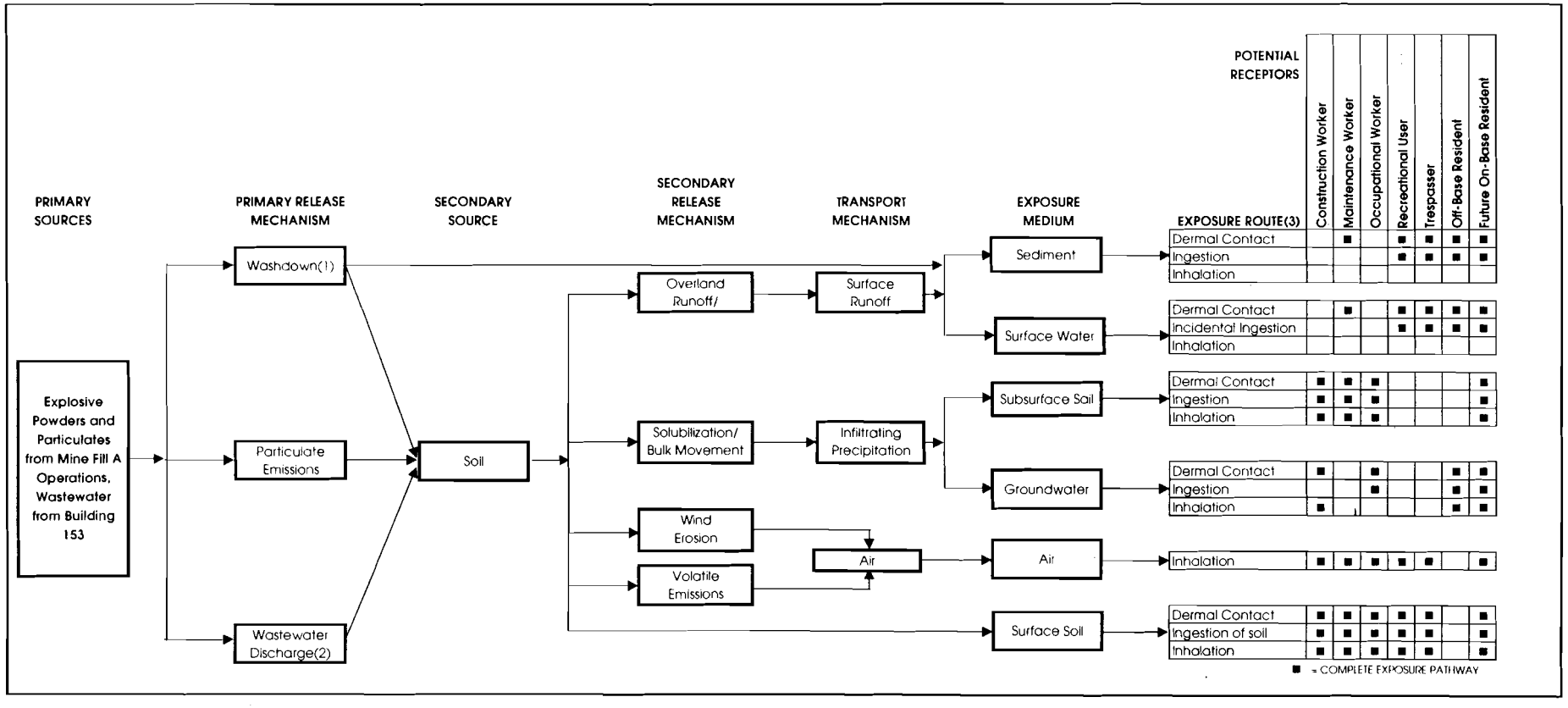
Receptor	Exposure Assumptions			Rationale/Reference
	Input Parameter	RME	CTE	
Resident (Adult/Small Child) (Continued)	IR_S (mg/day) [IR_{sed} (mg/day)]	100[1] [50[1]] 200[3] [100]	50[1] [25[1]] 100[3] [50]	Values for soil based on USEPA, May 1993. Values for sediment based on professional judgement.
	IR_a (m^3/day) (transfers from soil to air)	20[1] 8.3[3]	20[1] 8.3[3]	Adult daily inhalation rate (USEPA, December 1989) Child daily inhalation rate -- Recommended long-term rate for children, 3-5 years of age (USEPA, August 1997; Table 5-23).
	IR_a (m^3/hr) (showering)	0.6	0.6	For all age groups while showering (USEPA, December 1989).
	IR_w (L/day)	2[1] 1.5[3]	1.4[1] 0.66[3]	USEPA, May 1993 for adult exposure. USEPA, August 1997, Table 3-30 for child exposure.
	EV (events/day)	1	1	Professional judgement.

EF = Exposure Frequency
ED = Exposure Duration
ET = Exposure Time
BW = Body Weight
SA = Body Surface Area Exposed
IRs = Ingestion Rate - Soil
IRa = Inhalation Rate
EV = Exposure Events
CR = Contact Rate - Surface Water
Irw = Ingestion Rate - Groundwater
FI = Fraction soil/sediment ingested from source
IR_{sed} = Ingestion Rate - Sediment

1 Exposure assumption for adult receptor.
2 Exposure assumption for adolescent receptor.
3 Exposure assumption for small children (0 to 6 years of age).
4 Assumes domestic use of a ground water or surface water supply.
5 Assumes typical residential exposure to surface soils/sediments.

FIGURE C-1

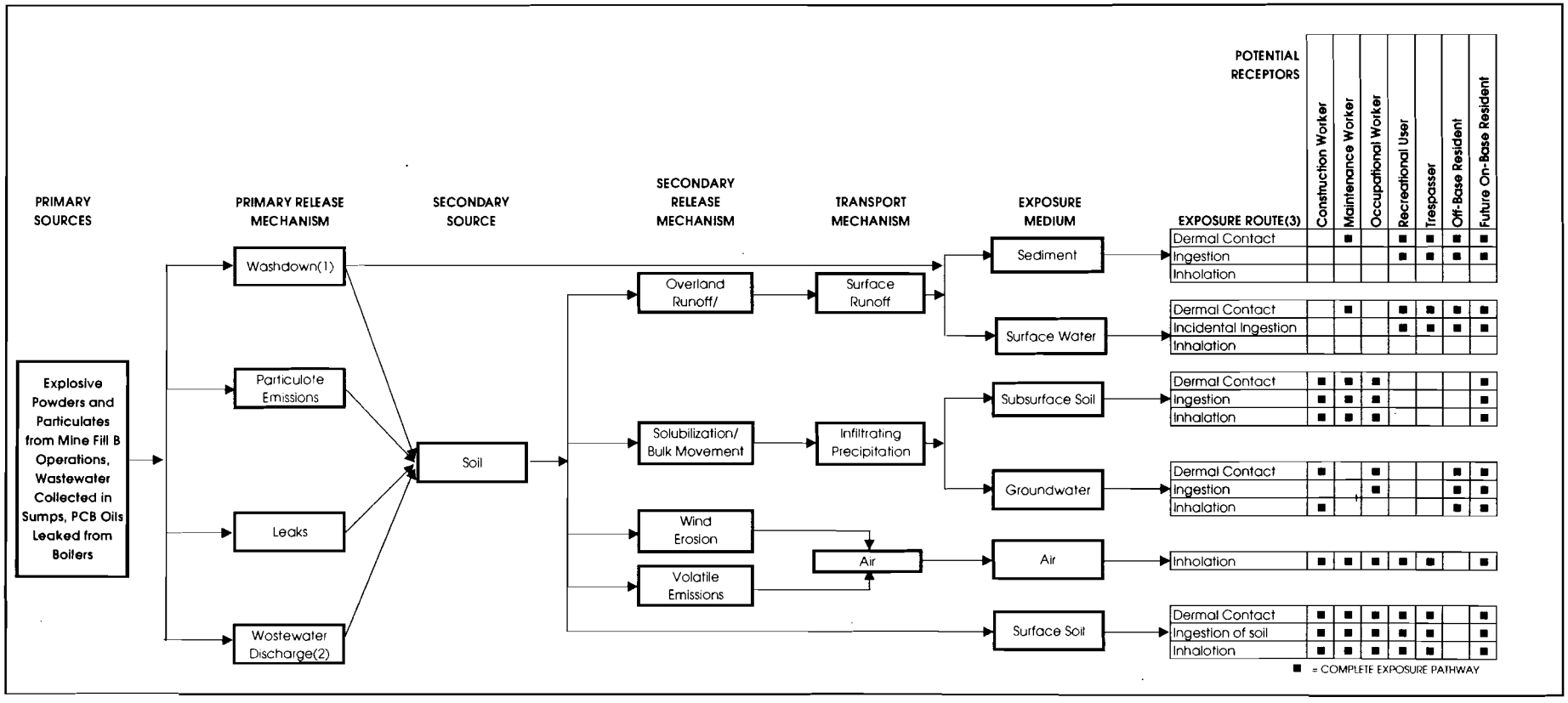
HUMAN HEALTH CONCEPTUAL SITE MODEL
SWMU 12 - MINE FILL A
NSWC CRANE, INDIANA



1. Washdown of powders and particulates from rooftops and the railroad lines near box houses, shaker rooms, and magazines.
2. Wastewater from Building 153 discharged to the settling pond from which contaminants may have percolated into groundwater. Wastewater also flowed from the pond to the north drain, potentially contaminating nearby surface water.
3. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

FIGURE C-2

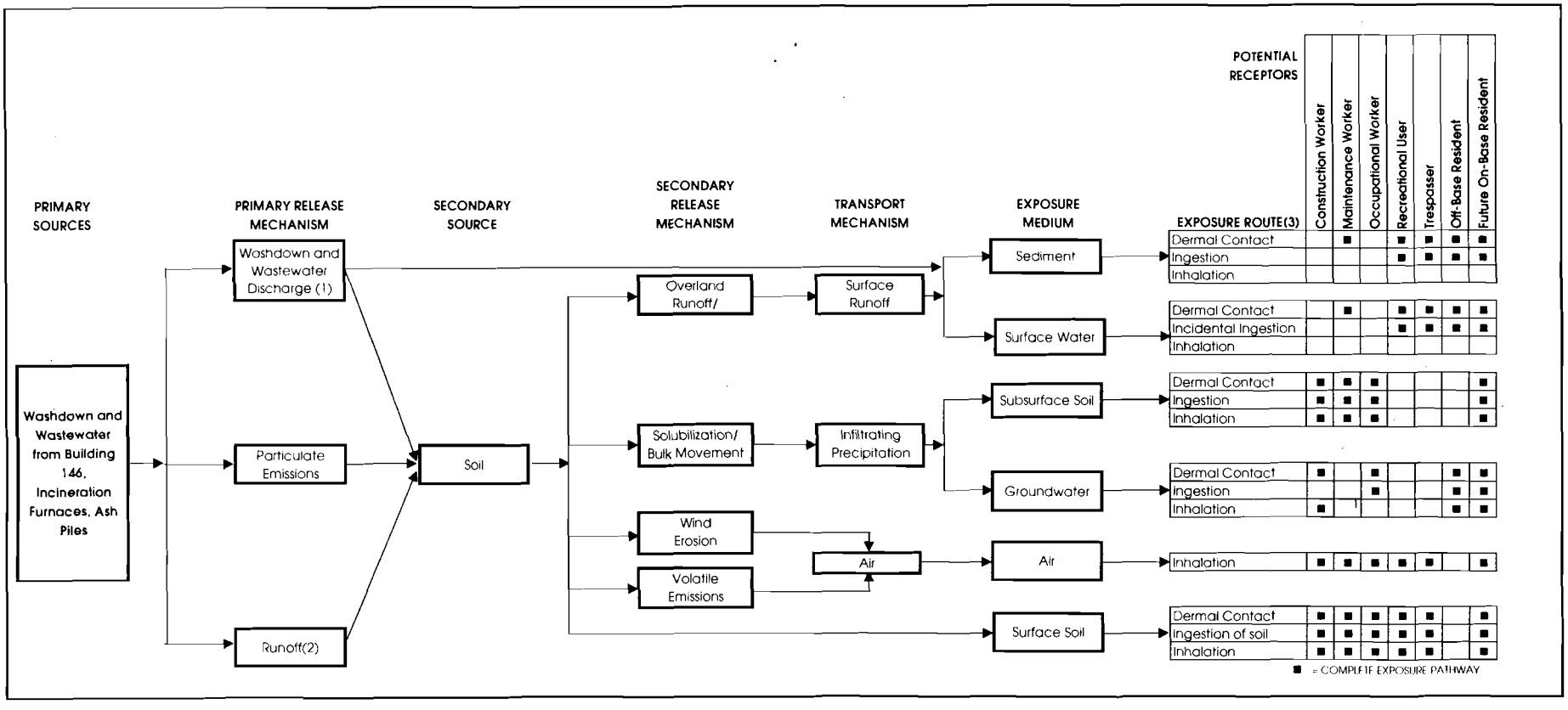
HUMAN HEALTH CONCEPTUAL SITE MODEL
SWMU 13 - MINE FILL B
NSWC CRANE, INDIANA



1. Washdown of powders and particulates from rooftops.
2. Wastewater containing explosives was collected in sumps which released the wastewater directly to surface drainage which flows into Boggs Creek.
3. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

FIGURE C-3

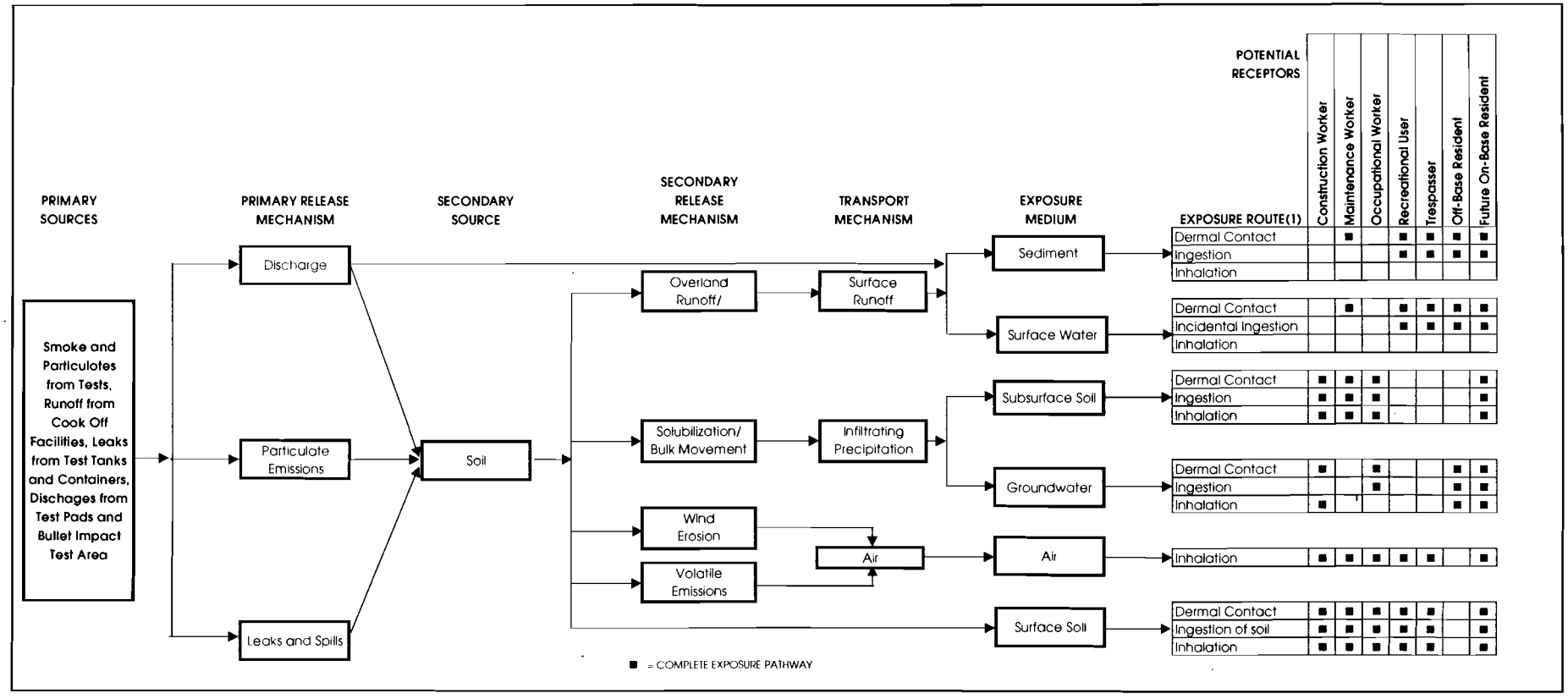
HUMAN HEALTH CONCEPTUAL SITE MODEL
SWMU 16 - CAST HIGH EXPLOSIVE FILL / INCINERATOR BUILDING 146
NSWC CRANE, INDIANA



1. Washdown and washout wastewater were discharged through floor drains of Building 146 to sumps which emptied into the Boggs Creek watershed.
2. Runoff from incinerator ash piles.
3. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

FIGURE C-4

HUMAN HEALTH CONCEPTUAL SITE MODEL
SWMU 19 - PYROTECHNIC TEST AREAS
NSWC CRANE, INDIANA



1. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

APPENDIX D

ECOLOGICAL RISK ASSESSMENT METHODOLOGY

APPENDIX D
ECOLOGICAL RISK ASSESSMENT METHODOLOGY

D.1 INTRODUCTION

The goal of the screening-level ecological risk assessment (SERA) to be performed as part of the RFI will be to identify the chemicals detected at concentrations that exceed the contaminants of potential concern (COPC) screening levels, the locations of these exceedances, and the need for further investigation and/or remedial action at the SWMUs 12, 13, 16, and 19 at NSWC Crane.

The SERA will provide information to scientists and managers that will enable them to conclude that either ecological risks for the study area are most likely negligible or that further information is necessary to better evaluate potential ecological risks. A phased approach to the SERA will be used that relies first on existing and then new environmental chemistry data for the preliminary assessments. Biological sampling or testing then may be conducted if further work is needed. However, such sampling and testing are not proposed at this time. The SERA methodology used at NSWC Crane will follow the guidance presented in the Final Guidelines for Ecological Risk Assessment (U.S. EPA, 1998) and the Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Ecological Risk Assessments (U.S. EPA, 1997a).

The SERA will consist of the first two of eight steps required by the United States Environmental Protection Agency (U.S. EPA) guidance (U.S. EPA, 1997 and 1998) and the Navy Policy for Conducting Ecological Risk Assessments (Navy, 1999). Figure D-1 presents the Navy's Ecological Risk Assessment Tiered Approach. The first two steps are the screening-level assessment. Step 3a is the first step of the baseline ecological risk assessment (BERA), which consists of refining the list of COPCs that were retained following the SERA. Steps 3b through 7 are conducted if additional evaluations or investigations are necessary. Finally, Step 8, Risk Management, is incorporated throughout the ERA process, in cooperation with Region 5 regulators.

The first phase in the ERA process is the screening-level risk assessment. In this phase, conservative exposure estimates are made for grouped or individual ecological receptors, and these exposures are compared to screening levels and threshold toxicity values. The SERA includes the following considerations:

- Screening-level problem formulation

- Screening-level ecological effects evaluation
- Screening-level exposure estimate
- Screening-level risk calculation

These sections are discussed in detail throughout this appendix.

D.2 SCREENING-LEVEL PROBLEM FORMULATION

The screening-level problem formulation includes identification of potential receptor groups, COPCs, and the mechanisms for fate and transport and toxicity. Determination of the complete exposure pathways that exist at a site is accomplished at this point to facilitate receptor selection. As part of receptor identification, site habitats and potential ecological receptors are described.

D.2.1 Environmental Setting

D.2.1.1 Basewide Environmental Setting

A biological characterization of NSWC Crane, including a list of plants and animals found at the facility, is presented in the Installation Assessment (IA; Army, 1978) and the Initial Assessment Study (IAS; NEESA, 1983) and is summarized in the Environmental Monitoring Reports (EMR; Halliburton NUS, August and November, 1992). A list of the species that may inhabit NSWC Crane and are protected under the U.S. Endangered Species Act, Indiana Department of Natural Resources Heritage Data Center, or the U.S. Fish and Wildlife Service is summarized in the RCRA Facility Permit (U.S. EPA, 1995). The following paragraphs briefly summarize the environmental setting at the base.

Eighty percent of NSWC Crane's 63,000 acres is classified as Central Hardwoods Forest of the United States (NEESA, 1983). In addition, some agricultural fields are in various stages of succession. Openings on dry upland sites contain almost pure stands of grasses with some clumps of woody plants such as persimmon, sassafras, and sumac. Wetter sites have river birch, willow, sycamore, and cottonwood. Hillside communities have mostly hickory, white and black oak, red maple, sugar maple, tulip poplar, ash, and beech (NEESA, 1983).

The great variety of habitats at NSWC Crane (i.e., many stages of forest succession, streams, ponds, Lake Greenwood, grassy open spaces) lead to a high animal species diversity (NEESA, 1983). Some of these species include (but are not limited to) mammals such as white-tailed deer, beaver, coyote, hawks,

red fox, rabbits, raccoons, and mice; birds such as ducks, geese, wild turkey, bobwhite quail, red-tailed hawks, and American robins; and various amphibians, reptiles, fish, and invertebrates.

The bird population includes a number of species that are state or federal threatened, endangered, or species of special concern and that use the site as their home range. These species include the bald eagle, osprey, sharp-shinned hawk, red-shouldered hawk, broad-winged hawk, black and white warbler, hooded warbler, and the worm-eating warbler (B&R Environmental, 1997).

The Indiana bat, a federal endangered species, is known to forage at the base. No Indiana bats were collected near SWMUs 12, 13, 16, or 19 during mist net surveys conducted at the base (Whitaker, 1996). Because of the bat and its potential habitat, the cutting of trees is restricted to certain times during the year, and the cutting of shagbark hickory trees is prohibited.

Six main creeks receive drainage in five separate drainage basins at NSWC Crane: Furst Creek, Sulphur Creek, Little Sulphur Creek, Boggs Creek, Turkey Creek, and Seed Tick Creek. There also are many smaller streams and creeks and drainage ditches located at the facility, along with several small man-made ponds and one large lake (Lake Greenwood). Lake Greenwood is the source of potable water for NSWC Crane. Surface water from the facility eventually discharges to the east fork of the White River, which is located south of the facility.

D.2.1.2 Site Specific Environmental Setting

This section presents the environmental setting at each SWMU. Figures showing each of the SWMUs locations are presented in Section 1.0 of the QAPP.

SWMU 12 – Mine Fill A

SWMU 12 consists of buildings, roads, railroad lines, parking lots, and grass-covered fields. The grass fields make up a large percentage of the SWMU area. There is little ecological habitat at the SWMU. Surface drainage at Mine Fill A is routed via ditches and culverts to intermittent drainageways transcending slopes on the eastern and western borders of the site. The swales are generally grass covered, and the media in these ditches could be considered either soil or sediment. Drainage from these ditches flows into larger intermittent drainageways, which flow south-southeastward into Turkey Creek. Turkey Creek flows south-southwestward to merge with Boggs Creek, which continues southward past the southern boundary of the base.

SWMU 13 – Mine Fill B

SWMU 13 consists of buildings, roads, railroad lines, parking lots, and grass-covered fields. The grass fields make up a large percentage of the SWMU area. There is little ecological habitat at SWMU 13. Surface drainage at Mine Fill B is routed via ditches and culverts to intermittent drainageways transcending slopes on the northwestern and southeastern boundaries of the site. The majority of surface drainage is collected and drained toward the northwest. Surface drainage flowing toward the northwest enters a southwestward-flowing intermittent tributary to Boggs Creek. Boggs Creek continues to flow southward past the southern boundary of the base. Surface drainage flowing down the slope southeast of Mine Fill B enters intermittent drainageways, which converge into two larger intermittent drainageways. These larger intermittent drainageways flow into Turkey Creek, which flows south-southwestward to merge with Boggs Creek.

SWMU 16 – Cast High Fill

Most of SWMU 16 is covered with buildings and gravel parking lots, with most grassy areas located southeast of Building 146. There is little ecological habitat at the SWMU.

Surface drainage from the upland area around Building 146 drains into the ravines to the west, south, and east. One drainage ditch near the southwestern corner of Building 146 collects drainage from the gravel-covered storage area along the western side of the building. Some frogs were observed in the sump near Building 146. This ditch collects and diverts surface drainage over the western slope.

Surface runoff from the site descends the adjacent slopes to enter intermittent drainageways in the bottoms of ravines west and east of Building 146. These intermittent ravine drainageways converge at a point south of Building 146 to form one of the upstream fingers of Turkey Creek. Turkey Creek flows south-southwestward to merge with Boggs Creek.

SWMU 19 – Pyrotechnic Test Area

The Pyrotechnic Test Area lies within the Boggs Creek Watershed. Based on the calculated 100-year floodplain for Boggs Creek, approximately 85 to 90 percent of the Ordnance Test Area (OTA) lies within the 100-year floodplain. Two intermittent drainageways flow to the west toward Boggs Creek from the eastern half of the OTA. A single intermittent drainageway enters Boggs Creek from the western half of the OTA. The lower areas located in the flood plain are frequently flooded due to poor drainage. Pools

and puddles of standing water were frequently seen in the lower areas throughout the site that may have been caused, in part, by heavy rains the night before the site visit.

The OTA consists of a number of areas that are different in regard to site-related activities, contamination, and habitat. The entire OTA is surrounded by woods consisting primarily of deciduous trees. The OTA consists of low-lying grassy areas and areas (slopes) of higher altitude. Boggs Creek has steep banks (about 5 to 10 feet high) and is lined with shrub/scrub type vegetation, mostly growing within the banks of the creek. There is evidence of various types of semi-aquatic animal life (e.g., herons, raccoons) in the low areas. Frogs and evidence of crustaceans (mounds) were frequently seen (or heard). Skeletal remains of crustaceans were found in the grass, indicating that they may have been eaten by other animals. Aquatic birds, such as herons, were seen on the site. Foxes, coyotes, raccoons, and bobcats are also known to inhabit the facility.

A meandering, intermittent, westward-flowing drainageway lies directly north of the Annex area. Another intermittent, westward-flowing drainageway lies 500 feet south of the Annex area.

A southward-flowing, intermittent drainageway lies west of the Rocket Range. This drainageway continues past the Rocket Range for approximately 2/3 mile before entering Lake Gallimore. Lake Gallimore is a surface water impoundment created by a conservation dam on Boggs Creek. It is not uncommon for the Rocket Range to flood to depths as great as 3 feet during rain events.

D.2.2 Contaminants, Ecotoxicity, and Fate and Transport

Based on historical site data and sampling, the following parameters are among the site-related chemical contaminants known to be present or potentially present in environmental media in SWMUs 12/13/16/19:

SWMU 12

- Explosives [e.g., 2,4,6-trinitrotoluene (TNT) and HMX] and their degradation products (e.g., 2-amino-4,6-dinitrotoluene).
- Metals (e.g., aluminum).

SWMU 13

- Explosives (e.g., TNT and HMX) and their degradation products (e.g., 2-amino-4,6-dinitrotoluene).
- Metals (e.g., aluminum).

- Polychlorinated biphenyls (PCBs) (from PCB oils leaked from therminol boilers).

SWMU 16

- Explosives (e.g., TNT and HMX) and their degradation products (e.g., 2-amino-4,6-dinitrotoluene).
- Metals (e.g., lead).
- PCBs (from PCB-contaminated fuels or materials).
- Petroleum hydrocarbons (from waste oils and fuels).

SWMU 19

- Explosives (e.g., TNT and HMX) and their degradation products (e.g., 2-amino-4,6-dinitrotoluene).
- Metals (e.g., lead).
- PCBs (from PCB-contaminated fuels or materials).
- Petroleum hydrocarbons (from waste oils and fuels).
- Dyes.

The following sections present a brief discussion regarding the toxicity, potential food chain and trophic transfer, and fate and transport properties of each class of contaminants potentially present within the study area.

D.2.2.1 Physical and Chemical Characteristics

Physical and chemical characteristics of contaminants may affect their mobility, transport, and bioavailability in the environment. These characteristics include bioaccumulation factors (BAFs), organic carbon partition coefficients, and octanol water partition coefficients. The following paragraphs discuss the significance of each factor.

The SERA will use plant and invertebrate bioaccumulation factors to predict contaminant loading in plants and invertebrates. The following list presents the source of the bioaccumulation factors that will be used in the SERA.

- Plant Bioaccumulation Factors - Organics: Toxicity and Chemical-Specific Factors Database (ORNL, 1998a).

- Plant Bioaccumulation Factors - Inorganics: Empirical Models for the Uptake of Inorganic Chemicals from Soil by Plants (ORNL, 1998b)

- Soil Invertebrate Bioaccumulation Factors - PCBs and Inorganics: Development and Validation of Bioaccumulation Models for Earthworms (Sample et al., 1998).

Some of the BAFs presented in the listed documents estimate the tissue concentrations in dry weight. These values must be converted to wet weight for use in exposure estimation in the food-chain models by multiplying the BAF by the proportion of dry matter content of the organism (Sample, et al., 1997). The following table presents the proportion of dry matter that will be used to adjust the BAFs, if necessary (Sample et al., 1997).

Food Type	Percent Water Content	Percent Dry Weight
Terrestrial Invertebrates (earthworms)	84	16
Terrestrial Plants (monocots-young grass)	70	30

Other plant, invertebrate, and small mammal bioaccumulation factors obtained from the literature also may be used for contaminants that are not listed in the above-referenced sources. Contaminants that do not have bioaccumulation factors will be assigned a default value of 1.

Biota-sediment accumulation factors (BSAFs) will be used to predict contaminant concentrations in fish tissue from contaminant concentrations in sediment. The BSAFs for the organic compounds will be obtained from The Incidence and Severity of Sediment Contamination in Surface Waters of the United States, Volume 1: National Sediment Quality Survey (U.S. EPA, 1997b) and other literature, as deemed appropriate. Fish BSAFs for metals are not available, so a default value of 1.0 will be used for the metals.

The organic carbon partition coefficient (K_{oc}) measures the tendency for a chemical to partition between soil or sediment particles containing organic carbon and water. This coefficient is important in the environment because it determines how strongly an organic chemical will bind to the organic carbon in the sediment.

The octanol/water partition coefficient (K_{ow}) is the ratio of a chemical concentration in octanol divided by the concentration in water. The octanol/water partition coefficient has been shown to correlate well with bioconcentration factors in aquatic organisms and with adsorption to soil or sediment.

The BAFs, K_{oc}s, and K_{ow}s for all the contaminants detected in the soil and sediment will be included in the SERA.

D.2.2.2 Metals

Many metals are found naturally in the surface water, sediment, and soil at various concentrations due primarily to chemical weathering and fallout from volcanoes. Most metals are toxic to aquatic (i.e., fish, invertebrates) and terrestrial (i.e., plants, invertebrates, vertebrates) ecological receptors at certain concentrations; some metals are toxic at lower concentrations than others. Also, different chemical forms of the metals may be more toxic than other forms. For example, hexavalent chromium is typically more toxic than trivalent chromium, and methylmercury is more toxic than inorganic mercury. In addition, the toxicity of several metals (cadmium, chromium, copper, lead, nickel, silver, and zinc) to aquatic receptors in freshwater systems decreases with increasing water hardness.

Only a portion of the total bulk concentration of metals in soils is bioavailable to ecological receptors. The uptake and accumulation of trace elements by plants are affected by several soil factors such as pH, Eh, clay content, organic matter content, cation exchange capacity, nutrient balance, concentration of other trace elements in soil, soil moisture, and temperature (Tarradellas et al., 1997). The bioavailability of the metals, however, is not known because there are other soil factors that influence uptake.

Many of these same factors also will influence the bioavailability of metals to invertebrates in sediment. One way to estimate the bioavailable portion of certain divalent metals (cadmium, copper, lead, nickel, and zinc) in sediment is to measure the amount of acid volatile sulfides (AVS) and simultaneously extracted metals (SEM) in a sediment sample. If the molar concentration of AVS is higher than the molar concentration of SEM, then all the SEM metals are expected to be unavailable to aquatic invertebrates and, therefore, nontoxic. AVS plays little or no role in determining interstitial water concentrations of metals in aerobic systems or those with low productivity (i.e., where the absence of organic carbon limits sulfate reduction) (Ankley et al., 1995) or when ingestion of sediments is the primary exposure route (Lee et al., 2000).

Of the 29 elements essential for plant growth, seven are micronutrients, including copper, iron, manganese, and zinc (Tarradellas et al., 1997). Also, the following metals may stimulate plant growth but are only essential for some plant species: aluminum, cobalt, nickel, sodium, selenium, and vanadium (Tarradellas et al., 1997). Finally, some elements such as lead, cadmium, and mercury are toxic elements with no known function in plant metabolism (Tarradellas et al., 1997).

ORNL (1998b) has calculated soil-to-plant uptake factors for several metals based on a compilation of various studies. Cadmium, mercury, selenium, and zinc were the only metals (except for calcium and potassium) with mean uptake factors greater than one (1.02 to 2.25). None of the metals (except for calcium and potassium) have median uptake factors greater than one. Arsenic, cadmium, mercury, nickel, selenium, and zinc were the only metals (except for calcium, magnesium, and potassium) with upper 90th percentile uptake factors greater than one (1.1 to 5) (ORNL, 1998b). This indicates that most metals will not biomagnify in plants. Finally, it is reported that, for arsenic, copper, nickel, and zinc, the plant-based food chain may be protected because the toxic concentrations of these metals in plants are higher than those for animals; cadmium and selenium are not toxic to plants at high concentrations and may be accumulated in plants at levels that may be toxic to animals (Cockerham and Shane, 1994). Other metals such as lead, cobalt, and mercury can enter the food chain via plant uptake, but to a lesser extent (Cockerham and Shane, 1994).

Cadmium appears to accumulate in most species of earthworms at greater levels than any other metal (Satchell, 1983). This is supported by the high mean soil-to-plant uptake factor of 17 for cadmium, compared to mean uptake factors of 5.7 (zinc), 5.2 (mercury), 4.5 (silver), and 3.3 (lead). The remaining metals (except potassium, sodium, and some radionuclides) had mean uptake factors of 1.8 or less. Cadmium, mercury, nickel, silver, and zinc are the only metals with median uptake factors greater than one. The upper 90th percentile uptake factors were 40.7, 20.6, 15.3, and 12.9 for cadmium, mercury, silver, and zinc. The remaining metals had upper 90th percentile uptake factors of 4.7 or less (Sample et al., 1998).

D.2.2.3 Semivolatile Organic Compounds

The most common semivolatile organic compounds that are found at naval facilities include polycyclicaromatic hydrocarbons (PAHs) and phthalates. PAHs are a diverse group of compounds consisting of two or more substituted and unsubstituted polycyclic aromatic rings formed by the incomplete combustion of carbonaceous materials. PAHs are ubiquitous in the modern environment and commonly are constituents of coal tar, soot, vehicle exhaust, cigarette smoke, certain petroleum products, road tar, mineral oils, creosote, and many cooked foods. PAHs also are released to the environment through natural sources such as volcano and forest fire emissions. However, most of the emissions result from anthropogenic sources, largely wood burning for homes. Vehicular emissions are another primary source of PAHs. Hazardous waste sites can be a concentrated source on a local scale. Examples of such sites include former manufactured gas sites (i.e., sources of coal tar) and abandoned wood

treatment plants (i.e., sources of creosote) [Agency for Toxic Substances and Disease Registry (ATSDR), 1989a].

PAHs are transferred from surface water by volatilization and sorption to settling particles. The compounds are transformed in surface water by photooxidation, chemical oxidation, and microbial metabolism (ATSDR, 1989a). In soil and sediments, microbial metabolism is the major process for degradation of PAHs (ATSDR, 1989a). Although PAHs accumulate in terrestrial and aquatic plants, many organisms are able to metabolize and eliminate these compounds. Vertebrates can readily metabolize PAHs, but lower forms (insects and worms) cannot metabolize PAHs as quickly. Food-chain uptake does not appear to be a major exposure source to PAHs for aquatic animals (ATSDR, 1989a).

Plants and vegetables can absorb PAHs from soils through their roots and translocate them to other plant parts such as developing shoots (Eisler, 1987). In general, however, PAHs are not readily taken up by plants because these compounds are strongly adsorbed onto soil organic particles and root uptake is very inefficient (Donker, et al., 1994). As such, lower molecular weight PAHs (which would be more water soluble) are absorbed by plants more readily than higher molecular weight PAHs. This is indicated by the low soil-to-plant uptake factors, which were calculated using the K_{ow} for the contaminants. Finally, many higher plants can catabolize benzo(a)pyrene and possibly other PAHs (Eisler, 1987).

PAHs vary substantially in their toxicity to aquatic organisms. In general, toxicity increases as molecular weight increases, with the exception of some high molecular weight PAHs that have low acute toxicity. Most species of aquatic organisms rapidly accumulate PAHs from low concentrations in the ambient medium. However, uptake of PAHs is highly species specific, it is higher in algae, mollusks, and other species that are incapable of metabolizing PAHs (Eisler, 1987). The ability of fish to metabolize PAHs may explain why benzo(a)pyrene is frequently not detected or is found at only very low levels in fish from environments heavily contaminated with PAHs (ATSDR, 1989a).

Phthalates are compounds that are used in production of plastics (ATSDR, 1993). Most phthalates are expected to sorb to soil or sediment particles after their release because of their high Log K_{oc} values (Howard, 1989). Some phthalates may bioconcentrate in aquatic organisms (Spectrum Laboratories, 1999; Howard, 1989; ATSDR, 1989a).

D.2.2.4 Pesticides

Pesticides are used to control pestiferous invertebrates and, therefore, they are toxic to many soil and aquatic invertebrates. In addition, many pesticides are toxic to higher trophic-level ecological receptors such as mammals and birds. For example, DDT compounds have been linked to eggshell thinning and subsequent decreased survival of several birds of prey (such as eagles and falcons). Other pesticides such as chlordanes, dieldrin, aldrin, endrin, and heptachlor also are very toxic to mammals and birds (Newell et al., 1987).

Organochlorine insecticides such as DDT, chlordane, aldrin, dieldrin, heptachlor, endosulfan, and endrin and their associated breakdown products generally degrade very slowly and tend to be soluble in lipids, which results in bioaccumulation and possible increases in concentrations through food webs (Newman, 1998). Pesticides have high Log K_{oc} values so they are expected to sorb strongly to soil and sediment particles when released to the environment. Consequently, these compounds are not easily displaced from their site of application, whether by runoff or leaching to groundwater. As a result, these compounds typically will not be taken up by plants.

DDT, DDE, and DDD are highly lipid soluble, which, combined with an extremely long half-life, results in bioaccumulation (ATSDR, 1989b). When present in ambient water, DDT and its metabolites are concentrated in freshwater and marine plankton, insects, mollusks, and other invertebrates and fish (ATSDR, 1989b). Because these organisms are part of the food chain, a progressive accumulation of residues may result in high levels of residues in organisms at the top of the food chain (ATSDR, 1989b). Moderate to significant bioconcentration in aquatic species has been reported for dieldrin, with bioconcentration factors (BCFs) ranging from 100 to 10,000 (Howard, 1991). Heptachlor also has been reported to bioconcentrate in aquatic species, with bioconcentration factors in fish up to approximately 20,000 (Howard, 1991).

Chlordane will bioconcentrate in both marine and freshwater species (ATSDR, 1989c). In living organisms, chlordane concentrations are usually highest in samples collected near areas where chlordane was used to control termites or other pests, in predatory species, and in tissues with high lipid content (Eisler, 1990). Food-chain biomagnification is low except in certain marine mammals (Eisler, 1990).

D.2.2.5 PCBs

The term polychlorinated biphenyls (PCBs) commonly refers to a variety of mixtures of individual biphenyl isomers, each consisting of two joined benzene rings and up to 10 chlorine atoms. Mixtures of these isomers are known by their commercial designation, Aroclor. This trade name is followed by a four-digit number; the first two numbers indicate the type of isomer mixture and the last two numbers indicate the approximate weight percent of chlorine in the mixture (U.S. EPA, 1985).

PCBs released into water adsorb to sediments and other organic matter. Typically, PCB concentrations are greater in the sediment and suspended material than are disolved in the water column. Substantial quantities of PCBs in aquatic sediments can act as an environmental reservoir from which PCBs may be released slowly over a long period of time (ATSDR, 1989d). For PCBs that exist in the dissolved state in water, volatilization becomes the primary fate process. PCBs have the capability to bioaccumulate and biomagnify (U.S. EPA, 1985).

Degradation of PCBs in the environment is dependent upon the degree of chlorination. Generally, the more chlorinated the PCB molecule, the more persistent it will be in the environment. Factors that determine biodegradability include the amount of chlorination, concentration, type of microbial population, available nutrients, and the temperature (ATSDR, 1989d).

PCBs are expected to be highly immobile in the soil due to rapid and strong sorption (ATSDR, 1989d). Some data indicate that plants are capable of taking up PCBs and transferring them into polar metabolites or insoluble molecules (Donker et al., 1994). However, it is not very probable that uptake and transformation of these compounds occur to any great extent, because a large part (greater than 95 percent) will adsorb to the root surface (Donker et al., 1994). The transfer of vapor-phase PCBs from air to aerial plant parts may be the main source of vegetation contamination (ATSDR, 1989d).

Because PCBs are highly lipophilic, they can bioaccumulate in the lipid portions of animals. Bioconcentration factors in the thousands have been reported for various aquatic species (Eisler, 1986). PCBs also can accumulate in upper trophic-level animals such as piscivorous birds and mammals that feed on contaminated prey items (Eisler, 1986). Finally, Sample et al. (1998) calculated mean, median, and 90th percentile reported soil-to-earthworm bioaccumulation factors (BAF) of 8.9, 6.7, and 15.9, respectively, indicating that PCBs can accumulate in soil invertebrates.

Adverse effects of PCBs on terrestrial wildlife include increased mortality, reproductive effects, and behavioral effects (U.S. EPA, 1985). As a group, birds are more resistant to acutely toxic effects of PCBs

than mammals (Eisler, 1986). Among sensitive avian species, PCBs disrupt the normal pattern of growth, reproduction, metabolism, and behavior (Eisler, 1986). Of the mammals, the mink is the most sensitive wildlife species tested for which data are available (Eisler, 1986). Impacts to mink include anorexia, weight loss, lethargy, reproductive effects, and death (Eisler, 1986).

D.2.2.6 Volatile Organic Compounds

VOCs are usually very mobile in the environment because they are poorly adsorbed to soil and sediment particles. Also, because they are very volatile, they typically are only detected in surface waters and surface soils at low concentrations.

Most VOCs have very little potential to bioaccumulate in ecological receptors; therefore, biomagnification through the food chain does not appear to be significant. VOCs are not expected to biomagnify in plants and are typically only toxic to ecological receptors only at relatively high concentrations.

D.2.2.7 Explosives

Some of the more common explosives include nitroaromatic compounds such as 2,4,6-trinitrotoluene, hexahydro-1,3,5-trinitro-1,3,5-triazine, octahydro-1,3,5,7-tetranitro-1,3,5-tetrazocine, N-methyl-N,2,4,6-tetranitroaniline, and associated by-products and degradation products that may have been released to the environment during manufacturing and load, assembly, and pack processes at military facilities (Talmage et al., 1999). These explosives are moderately to highly toxic to freshwater organisms, with chronic screening values less than 1 mg/L, although some of the screening values are low because of the conservative methods used to develop them based on an absence of data (Talmage et al., 1999). Available data indicate that none of the compounds are expected to bioconcentrate (Talmage et al., 1999). Most of the explosives do not appear to be highly toxic to mammals.

Explosives have little to moderate potential to adsorb to soil and sediment (Talmage et al., 1999). Therefore, explosives will have moderate to high mobility in soils and sediment, and most of the explosives will be found in the water column (Talmage et al., 1999).

D.2.3 Potential Exposure Pathways

Section 1.3 of the QAPP presents detailed descriptions of the potential sources of contamination for each of the SWMUs. The following bullets summarize the potential sources of contamination for the SWMUs, noting that the sources are not the same at each SWMU:

- Explosive powders and particulates
- Wastewater
- PCB oils from boilers
- Washdown water
- Incineration furnaces
- Ash piles

The contaminants from these sources may have migrated to soil, sediment, surface water, and/or ground water via various transport pathways (e.g., particulate emissions, runoff, leaks, direct discharge, etc.). Figures D-2 through D-5 present the conceptual site models (CSMs) for SWMUs 12, 13, 16, and 19, respectively. The following sections briefly discuss the CSMs for each SWMU.

Ecological receptors will not be directly exposed to contaminants in the groundwater at any of the SWMUs, so this exposure pathway is not complete. Exposure to groundwater after it discharges to a surface water body will be evaluated as part of the surface water pathway. Although, inhalation of particulates may be a complete pathway, it is expected to be insignificant compared to other pathways such as ingestion of food items that have accumulated contaminants from soil. Also, inhalation pathways typically are not evaluated in SERAs because of the uncertainty in exposures and effects concentrations. Therefore, this pathway will not be evaluated. Because most, if not all, of the food consumed by target receptors selected for this SERA will primarily be exposed to contaminants in the surface soil, exposure to subsurface soils will not be evaluated in the SERA. Finally, dermal exposure of terrestrial wildlife to contaminants in the soil is unlikely to represent a major exposure pathway because fur, feathers, and chitinous exoskeletons are expected to minimize transfer of contaminants across dermal tissue. Therefore, the dermal pathway will not be evaluated in the SERA.

D.2.3.1 SWMU 12

Historically, explosive powders and particulates were washed off roof tops and from the railroad line near box houses, shaker rooms, and magazines onto soil or directly into surface water or sediment. The railroad lines were hosed down once a day and, prior to the particulate abatement system, roofs were hosed off once a week. Also, wastewater from Building 153 was discharged to the settling pond and may have percolated into groundwater or discharged to the north drain, potentially contaminating nearby surface water. Based on the habitat at the site, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the surface soil.

The drainageways leading away from the site are intermittent and may be more representative of surface soil than true sediment. However, during wetter periods of the year, some of the drainageways may support benthic invertebrates. Also, contaminants from these drainageways may be transported downstream to waterbodies that can support a more stable benthic population. Therefore, sediment invertebrates, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the drainageways.

D.2.3.2 SWMU 13

Explosive powders and particulates from SWMU 13 were washed down from building rooftops onto soil or directly into surface water or sediment. Also, wastewater that collects in sumps may have percolated into groundwater or discharged to the surface drainage, potentially contaminating nearby surface water. Based on the habitat at the site, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the surface soil.

The drainageways leading away from the site are intermittent and may be more representative of surface soil than true sediment. However, during wetter periods of the year, some of the drainageways may support benthic invertebrates. Also, contaminants from these drainageways may be transported downstream to waterbodies that can support a more stable benthic population. Therefore, sediment invertebrates, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the drainageways.

D.2.3.3 SWMU 16

Contaminants from SWMU 16 were washed through floor drains of Building 146 into sumps. The wastewater may have percolated into groundwater or discharged to the surface drainage, potentially contaminating nearby surface water. Also, runoff from the incinerator ash piles may have transported contaminants to the surrounding soils and/or drainageways. Based on the habitat at the site, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the surface soil.

The drainageways leading away from the site are intermittent and may be more representative of surface soil than true sediment. However, during wetter periods of the year, some of the drainageways may support benthic invertebrates. Also, contaminants from these drainageways may be transported downstream to waterbodies that can support a more stable benthic population. Therefore, sediment

invertebrates, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the drainageways.

D.2.3.4 SWMU 19

Contaminants from SWMU 19 were deposited on the soil during various detonation activities, leaks, spills, and various discharges. Runoff from the detonation areas may have transported contaminants to the surrounding soils and/or drainageways. Based on the habitat at the site, plants, soil invertebrates, mammals, and birds may be exposed to chemicals that have been deposited in the surface soil.

The drainageways leading away from the site drain into Boggs Creek. The drainageways may support invertebrates or populations of small fish. However, Boggs Creek is known to support a larger fish population. Therefore, sediment invertebrates and small fish may be exposed to chemicals that have been deposited into the drainageways, and sediment invertebrates, fish, mammals, and birds may be exposed to chemicals that have been deposited into Boggs Creek.

D.2.4 Endpoints

D.2.4.1 Assessment Endpoints

Assessment endpoints are an explicit expression of the environmental value that is to be protected (U.S. EPA 1997a). The selection of these endpoints is based on the habitats present, the migration pathways of probable contaminants, and the routes that contaminants may take to enter receptors.

As already discussed in D.2.1.2, the habitat at and adjacent to the SWMUs consists primarily of open areas with grasses and shrubs surrounded by wooded areas. The drainageways adjacent to the sites are intermittent, but they ultimately drain to larger waterbodies. Based on the habitats at the SWMUs, the assessment endpoints are the protection of the following groups of receptors from adverse effects of contaminants on their growth, survival, and reproduction:

- Soil invertebrates
- Terrestrial vegetation
- Herbivorous mammals
- Herbivorous birds
- Soil invertebrate-eating birds
- Soil invertebrate-eating mammals

- Carnivorous birds
- Carnivorous mammals
- Omnivorous mammals
- Omnivorous birds
- Benthic invertebrates
- Fish
- Amphibians and reptiles

The following paragraphs discuss the reasons the above assessment endpoints were selected for this SERA.

Soil Invertebrates: Soil invertebrates are expected to be present in the soil at the site. They aid in the formation of soil and the redistribution and decomposition of organic matter in the soil, and they serve as a food source for higher trophic-level organisms. They also can accumulate some contaminants that can then be transferred to the higher trophic-level organisms that consume invertebrates.

Terrestrial Vegetation: Terrestrial vegetation at the site consists of grasses, shrubs, and trees. They serve as a food source and provide shade and cover for many organisms, and they help prevent soil erosion, among other important functions. They also can accumulate some contaminants that can then be transferred to the higher trophic-level organisms that consume plants.

Herbivorous Birds and Mammals: Herbivorous birds and mammals (animals that consume only plant tissue) may be present at the site because of the vegetative habitats (i.e., forested). Their role in the community is essential because, without them, higher trophic levels could not exist (Smith, 1966). They may be exposed to and accumulate contaminants that are present in the plants they consume.

Carnivorous Birds and Mammals: Carnivorous birds and mammals consist of birds and mammals that consume invertebrates, fish, and other mammals and birds. Soil invertebrate-eating birds and mammals are present throughout the base in different terrestrial habitats (i.e., forested, open field). These are considered first-level carnivores and they serve as a food source for higher trophic-level carnivores. Piscivorous birds and mammals may be present along Little Sulfur Creek based on the presence of fish in the creek (in the spring-feed portion of the creek below the Jeep Trail). Finally, carnivorous birds and mammals that feed on other birds and mammals are at the top of the food chain. The top carnivores typically are less densely distributed than the herbivores and first-level carnivores because they require a

larger area to hunt for their food. All the carnivores may be exposed to and accumulate contaminants that are present in the food items they consume.

Omnivorous Birds and Mammals: Omnivorous birds and mammals (that consume both plant and animal tissue) are present throughout the base in the different terrestrial habitats (i.e., forested, open field). They may be exposed to and accumulate contaminants that are present in the plants and animals they consume.

Benthic Macroinvertebrates: Benthic macroinvertebrates are similar to the soil invertebrates in that they serve as a food source for higher trophic-level organisms (i.e., fish, amphibians, birds, mammals). They also can accumulate some contaminants that can then be transferred to the higher trophic-level organisms that consume invertebrates. Their presence in the intermittent drainageways will be ephemeral because of a lack of suitable habitat and water flow.

Fish: Fish will not be present in the drainageways leading away from the sites because the drainageways are intermittent and usually dry. However, fish may be present in the downstream waterbodies from the intermittent ditches. Also, fish are present in Boggs Creek adjacent to SWMU 19 and may be present in some of the drainageways from SWMU 19 to Boggs Creek. Fish are exposed to and can accumulate contaminants from the food items they consume or from the surface water in which they live.

Amphibians and Reptiles: Amphibians are expected to inhabit water bodies and the surrounding areas, and reptiles can inhabit both aquatic environments and terrestrial habitats. Amphibians and reptiles feed primarily on invertebrates, plants, fish, and/or small mammals. They are exposed to and can accumulate contaminants from the food items they consume or from the surface water, sediment, or surface soil in which they live.

D.2.4.2 Measurement Endpoints

All the above assessment endpoints will not be evaluated in the SERA for reasons presented below. As indicated in U.S. EPA (1997a), "it is not practical or possible to directly evaluate risks to all of the individual components of the ecosystem at a site. Instead, assessment endpoints focus the risk assessment on particular components of the ecosystem that could be adversely affected by contaminants from the site." Therefore, the SERA will focus on the endpoints that will tend to yield the highest risks, which should then account for endpoints that will have lower risks.

Measurement endpoints are estimates of biological impacts (e.g., mortality, growth and reproduction) that are used to evaluate the assessment endpoints. The following measures of effects will be used to evaluate the assessment endpoints in this ERA, where applicable.

- Soil screening values – Mortality, growth, and reproduction of plants and soil invertebrates will be evaluated by comparing the measured concentrations (maximum and average) of chemicals in the surface soil to screening values designed to be protective of ecological receptors.

- No-observed-adverse-effects levels (NOAELs) for surrogate wildlife species - Mortality, reproductive, and/or developmental effects of birds and mammals will be evaluated by comparing the estimated ingested dose (based on conservative and average assumptions) from contaminants in the surface water, sediment, surface soil, plants, invertebrates, and fish to these levels.

- Sediment screening values – Mortality and other adverse effects (e.g., growth, feeding rates, behavioral changes) of benthic macroinvertebrates will be evaluated by comparing the measured concentrations (maximum and averages) of chemicals in the sediment to screening values designed to be protective of ecological receptors.

- Surface water screening values – Mortality and other adverse effects (e.g., growth, feeding rates, behavioral changes) of aquatic organisms will be evaluated by comparing the measured concentrations (maximum and averages) of chemicals in the surface water to screening values designed to be protective of ecological receptors.

D.2.4.3 Selection of Receptor Species

Many receptors in the soil and aquatic environments are adequately described in general categories such as soil invertebrates, vegetation, and sediment-dwelling (benthic) invertebrates. This is due to the nature of the threshold values, effects values, or water-quality criteria that are typically used to characterize risk for such organisms. For vertebrate receptors, selection of particular species may be required so that intake through eating, drinking, and other routes can be estimated.

Receptor identification is influenced by the contaminants, their likely mode of transport, ultimate fate, and toxicity. For example, most metals (with notable exceptions of cadmium and mercury) typically do not bioaccumulate. For contaminants that bioaccumulate, such as mercury compounds and chlorinated pesticides, effects on upper trophic-level receptors need to be assessed. For contaminants that do not

bioaccumulate, organisms that are in direct contact with soil/sediment (i.e., sediment- and soil-dwelling organisms and plants) and animals that may incidentally ingest soil particles are selected as receptors for metals if exposure pathways are complete. Sensitivity to particular contaminants is also considered. For example, birds and mammals may have different sensitivities to organic compounds, so each group, or the most sensitive group for a particular contaminant, is assessed.

As previously mentioned, for most receptor species, ingestion is the primary route of exposure. Indicator species are selected for their preferred habitat, body size, sensitivity, home range, abundance, commercial or sport utilization, legal status, and functional role (e.g., predators). For conservativeness, indicator species may be small and have small home ranges. Species known to be sensitive to particular contaminants may be selected, or toxicity values for those species may be used. For example, mink are sensitive to PCBs for reproductive endpoints and therefore mink TRVs would be selected for a scenario involving exposure to PCBs from an aquatic or sedimentary source. The availability of exposure parameters such as body mass, feeding rate, and drinking rate may also be a factor in selecting indicator species. The following indicator species will be used for the food-chain modeling (discussed later):

- Herbivorous mammal - meadow vole (SWMUs 12, 13, 16, and 19)
- Herbivorous bird - bobwhite quail (SWMUs 12, 13, 16, and 19)
- Carnivorous bird - american robin (SWMUs 12, 13, 16, and 19)
- Carnivorous mammals - short tail shrew (SWMUs 12, 13, 16, and 19)
- Piscivorous mammal - raccoon (SWMU 19)
- Piscivorous bird - belted kingfisher (SWMU 19)

Receptor profiles for each of these species are presented in Attachment D-1.

D.3 ECOLOGICAL EFFECTS EVALUATION

The preliminary ecological effects evaluation is an investigation of the relationship between the magnitude of exposure to a chemical and the nature and magnitude of adverse effects resulting from exposure. In addition to being a toxicity study, it may also include descriptions of apparent effects seen during the site visit. Toxicity thresholds are usually expressed in units of concentration when the medium of concern is in intimate contact with the receptor, such as surface water for pelagic organisms or soil for soil invertebrates. For other receptors, such as terrestrial vertebrates, toxicity data are typically available as doses, with units equal to mass of contaminant per unit of body mass per unit of time (usually mg/kg-day).

As the first step in the ecological effects evaluation, COPCs will be selected by comparing the contaminant concentrations in the surface water, sediment, and surface soil samples to Region 5 ecological data quality levels (EDQLs) (U.S. EPA, 1999a). Calcium, magnesium, potassium, and sodium will not be retained as COPCs in any medium because of their relatively low toxicity to ecological receptors and their high natural variability in concentrations. Contaminants without EDQLs will be retained as COPCs but they may only be evaluated qualitatively. The following procedures will be used in the SERA to select COPCs.

Surface Water and Sediment for Benthic Macroinvertebrates, Fish, and Terrestrial Wildlife

1. Inorganic and organic contaminants whose maximum concentrations do not exceed EDQLs will not be retained as COPCs.

2. Inorganic contaminants whose maximum concentrations do not exceed the maximum upstream or upgradient concentrations will not be retained as COPCs.

It is recognized that the EDQLs were not established for the protection of wildlife ingesting water. However, based on the very low and conservative EDQLs for surface water, contaminants that do not exceed EDQLs are not expected to be toxic to terrestrial wildlife.

Surface Soil for Invertebrates, Plants, and Terrestrial Wildlife

1. Inorganic and organic contaminants whose maximum concentrations do not exceed EDQLs will not be retained as COPCs.

2. Inorganic contaminants whose maximum concentrations do not exceed the site-specific background concentrations will not be retained as COPCs.

Contaminants that are retained as COPCs will be further evaluated as part of Step 3a of the eight-step ERA process. The next section presents the additional data sources that will be used to evaluate the COPCs.

D.4 STEP 3A – REFINEMENT OF COPCs

Step 3a consists of refining the list of COPCs from the SERA using less conservative screening values and more site-specific exposure assumptions (where available) to more realistically estimate potential

risks to ecological receptors (i.e., plants, invertebrates, and aquatic receptors). Note that the Step 3a evaluation will be included as an attachment to the SERA. This evaluation may include (but is not necessarily limited to) a consideration of the following topics:

- Magnitude of criterion exceedance: Although risks may not relate directly to the magnitude of a criterion exceedance, the magnitude may be one item used in a lines-of-evidence approach to determine the need for further site evaluation.

- Frequency of chemical detection: A chemical that is detected at a low frequency typically will be of less concern than a chemical detected at higher frequency if toxicity and concentrations of the constituents are similar. All else being equal, chemicals detected frequently will be given greater consideration than those detected relatively infrequently.

- Contaminant bioavailability: Many contaminants (especially metals) are present in the environment in forms that are typically not bioavailable, and the limited bioavailability will be considered when evaluating the exposures of receptors to site contaminants.

- Habitat: Although exceedances of criteria may occur, potential risks to ecological receptors may be minimal if there is little habitat for those receptors. Therefore, the extent of habitat will be used qualitatively when considering the site for additional evaluation.

D.4.1 Alternate Benchmarks

The following sections present some of the alternate benchmarks and evaluations that will be conducted as part of Step 3a.

D.4.1.1 Terrestrial Plants and Invertebrates

Risks to terrestrial plants and invertebrates resulting from exposure to the COPCs will be evaluated by comparing the contaminant concentrations in the surface soil to alternate soil benchmark values. These alternate benchmarks will be designated as surface soil screening levels (SSSLs). Currently, neither Indiana nor U.S. EPA has developed final ecological SSSLs. The following list presents the SSSLs that have been developed by a few groups or agencies. Additional details explaining the origin and basis for the alternate benchmarks are provided below the list.

- Dutch Intervention Values and Target Values – Soil Quality Standards (MHSPE, 2000).

- Canadian Soil Quality Guidelines (CCME, 1997).

- Oak Ridge National Laboratory Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision (R.A. Efroymson et al., 1997a).

- Oak Ridge National Laboratory Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision (R.A. Efroymson et al., 1997b).

The Intervention Values and Target Values – Soil Quality Standards were developed by the Netherlands Ministry of Housing, Spatial Planning, and Environment, Department of Soil Protection and will be referred to as the Dutch Screening Values (MHSPE, 2000). The Dutch Screening Values for surface soil consist of target values and intervention values. The target values are the soil-quality levels at which there is sustainable soil quality (MHSPE, 2000). The values for heavy metals, arsenic, and fluoride were derived from analysis of field data from relatively pollution-free rural areas. The intervention values indicate the concentration levels of the contaminants in the soil above which the functionality of the soil for human, plant, or animal life is seriously impaired or threatened (MHSPE, 2000). The target value will be used to determine ecological effects and the need for additional evaluation of the data.

The Canadian Soil Quality Guidelines were developed by the Canadian Council of Ministers of the Environment (CCME, 1997). They are derived using toxicological data to determine the threshold level for key receptors (CCME, 1997). The values are calculated for four land uses: agricultural, residential or parkland, commercial, and industrial. Exposure from direct soil contact is used to derive guidelines for the residential or parkland, commercial, and industrial land uses (CCME, 1997). However, the soil guidelines for the agricultural land use incorporate direct soil contact as well as soil and food ingestion (CCME, 1997). A more detailed discussion of the derivation of the soil quality guidelines is presented in A Protocol for the Derivation of Environmental and Human Health Soil Quality Guidelines (CCME, 1996).

The Toxicological Benchmarks for Contaminants of Potential Concern for Effects on Soil and Litter Invertebrates and Heterotrophic Process: 1997 Revision (Efroymson R.A., et al., 1997a) and the Toxicological Benchmarks for Screening Contaminants of Potential Concern for Effects on Terrestrial Plants: 1997 Revision (Efroymson R.A. et al., 1997b) were developed by the Oak Ridge National Laboratory (ORNL). These benchmarks were intended to be used as screening values and, as such,

may be overly conservative. They are based on a 20 percent reduction in growth, reproduction, or activity (for invertebrates) or growth and yield (for plants) as the threshold for significant effects (Efroymson et al., 1997a, b).

Additional sources of toxicity data from the literature may be used to evaluate potential risks to soil flora and invertebrates from contaminants in the surface soil for contaminants that are not evaluated in the above documents.

D.4.1.2 Surface Water

Water quality standards (WQS) for surface water have been developed for Indiana (IDEM, 1998). These are the primary enforceable standards for surface water at the SWMUs. In addition, U.S. EPA has established Ambient Water Quality Criteria (AWQC) for several contaminants. Other, non-regulatory surface water screening values will be used to evaluate the surface water data that do not have WQS or AWQC. All values will be collectively referred to as surface water screening levels (SWSLs) in this SERA QAPP. The following presents the SWSLs that will be used in this evaluation.

- Indiana Water Quality Standards (IDEM, 1998).
- Recommended Water Quality Criteria (U.S. EPA, 1999).
- Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects on Aquatic Biota, 1996 Revision (Suter and Tsao, 1996).
- Eco Update-Ecotox Thresholds (U.S. EPA, 1996).

The Indiana WQS are the concentrations of toxic substances that will not result in acute or chronic toxicity to aquatic life. All the WQS used in this SERA will be based on total recoverable metals in accordance with the Indiana WQS (IDEM, 1998).

The recommended water quality criteria were developed by U.S. EPA to provide states with guidance for developing their own criteria (U.S. EPA, 1999). These values are set to protect the majority of aquatic organisms from adverse impacts from contaminants in the surface water.

The publication ECO Update-Ecotox Thresholds was prepared by U.S. EPA for use as benchmark screening values in the first step of a baseline risk assessment (U.S. EPA, 1996). Most of the surface water thresholds for the contaminants that are evaluated in this SERA are based on Suter and Mabrey (1994) in the Ecotox Thresholds. Because Suter and Mabrey (1994) has been updated, Suter and Tsao

(1996) values will be used when the Ecotox Thresholds were based on the Suter and Mabrey (1994) data. The Suter and Tsao (1996) benchmarks were calculated using Tier II methodology, as described in the U.S. EPA's Proposed Water Quality Guidance for the Great Lakes System (U.S. EPA, 1993a). Tier II values are developed so that aquatic benchmarks can be established with fewer data than are required for the U.S. EPA AWQC.

Both the acute and chronic SWSLs will be used to evaluate the COPCs to determine potential impacts in a lines-of-evidence approach.

D.4.1.3 Sediment

Indiana has not established sediment-screening levels (SdSLs) for any contaminants, and U.S. EPA has established SDSLs for only a few contaminants. Therefore, other non-regulatory alternate benchmarks will be used to evaluate the sediment data. SdSLs based on freshwater studies will be used where available. The following list presents the SdSLs that will be used in this evaluation. The paragraphs following this list discuss the SdSLs in more detail:

- Eco Update-Ecotox Thresholds (U.S. EPA, 1996).
- Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario (OMOE, 1993).
- Incidence of Adverse Biological Effects within Ranges of Chemical Concentrations in Marine and Estuarine Sediments (Long et al., 1995).

The sediment Ecotox Thresholds include draft U.S. EPA sediment quality criteria (SQC) that have been established for five contaminants (acenaphthene, dieldrin, endrin, fluoranthene, and phenanthrene), sediment quality benchmarks (SQB) that have been established using equilibrium partitioning, and effects range-low values from Long et al. (1995). The SQC and SQBs Ecotox Thresholds are based on an assumption of one percent organic carbon [10,000 mg/kg total organic carbon (TOC)]. The three PAH SQC documents (acenaphthene, fluoranthene, and phenanthrene) have been withdrawn in favor of a draft PAH Mixture Sediment Guideline (U.S.EPA, 2000). In addition, the draft Sediment Guidelines for Metals (cadmium, copper, lead, nickel, silver, and zinc) was published by U.S. EPA (U.S. EPA, 2000). These documents will be utilized if they are finalized when the SERA is prepared.

The Guidelines for the Protection and Management of Aquatic Sediment Quality in Ontario (OMOE, 1993) are based on freshwater studies. The Ontario Ministry of Environment and Energy (OMOE) guidelines establish three effects levels, as follows:

- ***No-Effect Level:*** Sediment will not affect fish or sediment-dwelling organisms. In addition, no transfer through the food chain and no effect on water quality is expected.

- ***Lowest-Effect Level:*** Sediment is considered marginally polluted but will not affect the majority of sediment-dwelling organisms.

- ***Severe-Effect Level:*** Sediment is considered highly polluted and likely to affect the health of sediment-dwelling organisms.

The Incidence of Adverse Biological Effects within Ranges of Chemical Concentrations in Marine and Estuarine Sediments (Long et al., 1995) will be used if no freshwater data are available because they are generally accepted by many state agencies and U.S. EPA regions, even though they are based primarily on estuarine and marine studies. Long et al. (1995) establishes three effects levels, as follows:

- ***Below Effects Range-Low (ER-L):*** (Effects Range-Low) Minimal-effects range (adverse effects would be rarely observed).

- ***Between ER-L and Effects Range-Median (ER-M):*** Possible-effects range (adverse effects would occasionally occur).

- ***Above the ER-M:*** Probable-effects range (adverse effects would probably occur).

Additional sources of toxicity data from the literature may be used to evaluate potential risks to aquatic receptors from contaminants in the sediment for contaminants that are not evaluated in the above documents. Finally, the AVS minus the SEM results will be used to determine if the metals included in the SEM analysis are bioavailable. Note that only true sediment samples (i.e., those sediments inundated with water for 30 consecutive days in a year) will be analyzed for AVS/SEM.

Contaminants that exceed the SdSLs also will be compared to background contaminant levels developed in the Sediment Background Concentration Distributions of 172 Potential Pollutants in Indiana (Wente, 1994) document. The term "background" was interpreted in that document as "the concentration that would be present in the absence of any particular pollutant source." Background values will be used as another piece of information in the lines-of-evidence approach for evaluating the sediments.

D.4.2 Food-Chain Modeling

Most of the above-mentioned standards and benchmark values are not designed to screen out risks to terrestrial wildlife ingestion of the soil, sediment, surface water, plants, invertebrates, and fish. Therefore, a terrestrial intake model will used to estimate the exposure of the COPCs to terrestrial receptors.

Risk to terrestrial receptors to the COPCs in the soil, sediment, and surface water will be determined by estimating the chronic daily intake (CDI) (see Section D.4.3) and comparing the CDI to terrestrial reference values (TRVs) representing acceptable daily doses in mg/kg-day. The TRVs will be developed from no-observed-adverse-effect-levels (NOAELs) and lowest-observed-adverse-effect-levels (LOAELs) obtained from wildlife studies, if available. The majority of the TRVs will come from the ORNL Toxicological Benchmarks for Wildlife: 1996 Revision (Sample et al., 1996). Toxicity data in the Agency for Toxic Substances and Disease Registry (ATSDR) toxicity profiles and IRLS printouts will be utilized, when necessary.

For avian species, the NOAEL (or LOAEL) for the test species will be used as the NOAEL (or LOAEL) for the surrogate species in accordance with Sample et al. (1996). For mammalian species, the NOAEL (or LOAEL) from one species will be adjusted to a NOAEL (or LOAEL) for the surrogate species using the following body weight scaling equation (from Sample et al., 1996):

$$NOAEL_w = NOAEL_t * (bw_t/bw_w)^{1/4}$$

Where: $NOAEL_w$ = no-observed-adverse-effect level for the surrogate wildlife species
$NOAEL_t$ = no-observed-adverse-effect level for the test species
bw_t = body weight of the test species
bw_w = body weight of the surrogate test species

The body weight scaling is performed because studies have shown that, for mammals, numerous physiological functions such as metabolic rate, as well as responses to toxic chemicals, are a function of body size (Sample et al., 1996). However, Sample et al. (1996) indicated that physiological scaling factors may not be appropriate for birds. Therefore, a scaling factor of 1.0 will be used for birds in this SERA.

Table D-1 presents the body weights that will be used for the surrogate and potential test species. If a subchronic study is used to develop the TRV, the final value will be multiplied by a factor of 0.1 to account

for uncertainty between subchronic and chronic effects. Also, if a LOAEL study is used to develop the NOAEL TRV, then the LOAEL will be multiplied by a factor of 0.1 to obtain the NOAEL.

D.4.3 Characterization of Exposure

This section describes the potential or actual contact or co-occurrence of the contaminants with the receptors to determine their exposure dose.

Terrestrial soil invertebrates and plants are exposed to contaminants in the surface soil through direct contact and/or ingestion. Aquatic organisms are exposed to contaminants in the surface water and sediment through direct contact and/or ingestion. The 95 percent Upper Confidence Level (UCL) and average soil, surface water, and/or sediment concentrations will be compared to the applicable soil screening values to determine potential risk to these ecological receptors to obtain a range of exposures.

Exposure of the terrestrial receptors to the COPCs in the surface soil, sediment, and surface water will be determined by estimating the daily doses in mg/kg-day using exposure equations. The contaminant concentrations in the surface soil, sediment, and surface water will be used to calculate the CDI doses. The following equation presents a generic food-chain model for chronic daily intake (CDI) that will be used for the surrogate species that are selected for modeling:

$$\text{CDI Dose (mg/kg - day)} = \frac{(FI * FC) + (WI * SW) + (SI * SC)}{BW}$$

Where:

	FI	= Food ingestion rate (kg/day)
	FC	= Food concentration (mg/kg)
	WI	= Surface water ingestion rate (L/day)
	SW	= Surface water concentration (mg/L)
	BW	= Body weight (kg)
	SI	= Incidental sediment ingestion rate (kg/day)
	SC	= Sediment concentration (mg/kg)

For inorganic constituents in surface soil and sediment, as well as organic constituents in surface soil, the contaminant concentration of the prey items (i.e., earthworms, fish) is calculated using the following equation:

$$FC = SC * BAF$$

Where: FC = Contaminant concentration in food
SC = Contaminant concentration in surface soil or sediment
BAF = Bioaccumulation factor

For organic constituents in the sediment, the contaminant concentration of the prey items is calculated using the following equation:

$$FC = \frac{SC * \%L * BSAF}{\%TOC}$$

Where:
FC = Contaminant concentration in food
SC = Contaminant concentration in sediment
%L = Percentage of lipids in fish [3.6% = the average of various species of sunfish. Attachment D-1 presents the calculation used to derive this value (U.S. EPA, 1997)]
BSAF = Biota-sediment accumulation factor (chemical-specific)
%TOC = Percentage of total organic carbon in sediment (site-specific)

The lower bound of the threshold effects is based on consistently conservative assumptions and NOAEL toxicity values (U.S. EPA, 1997a). This bound will present the highest potential risks. The upper bound is based on observed impacts or predictions that ecological effects could occur and is developed using consistent assumptions, site-specific data, LOAEL toxicity values, or an impact evaluation (U.S. EPA, 1997a). This bound will present the average potential risk. Both the upper and lower bounds will be evaluated in this SERA to provide the overall range of potential risks, as presented in the following table:

Conservative Scenario	Average Scenario
95% UCL soil, surface water, or sediment concentration.	Average soil, surface water, or sediment concentration.
90% BAF/BSAF value from the literature (when available).	Median BAF/BSAF value from the literature (when available).
Highest receptor body weight for NOAEL calculation.	Average receptor body weight for LOAEL calculation.
Lowest receptor body weight for CDI equation.	Average receptor body weight for CDI equation.
Conservative receptor ingestion rate.	Average receptor ingestion rate.
Use NOAELS.	Use LOAELs.
Receptors spend 100% of their time at the site.	Receptor's home range taken into account.

The exposure assumptions (i.e., ingestion rate, body weight) will be obtained from the Wildlife Exposure Factors Handbook (U.S. EPA, 1993b) or other literature sources, if necessary. Table D-1 presents the exposure parameters that will be used in the SERA. Table D.A-1 in Attachment D-1 presents the values that were used to calculate the exposure parameters and a discussion of how they were calculated.

D.5 ECOLOGICAL RISK CHARACTERIZATION

The risk characterization is the final phase of a risk assessment that compares the exposure to the ecological effects. It is at this phase that the likelihood of adverse effects occurring as a result of exposure to a stressor will be evaluated. An ecological effects quotient (EEQ) approach will be used to characterize the risk to terrestrial receptors. This approach characterizes the potential effects by comparing exposure concentration with the effects data. An EEQ of greater than 1.0 is considered to indicate a potential risk. The EEQ is not an expression of probability, and the meaning of values greater than 1.0 must be interpreted in light of attendant uncertainties in risk management.

An EEQ for the aquatic receptors will be calculated as follows:

$$EEQ = \frac{C_{sw}}{SWSL} \text{ or } \frac{C_{sd}}{SdSL}$$

Where:
- EEQ = Ecological effects quotient (unitless)
- C_{sw} = Contaminant concentration in surface water (µg/L)
- C_{sd} = Contaminant concentration in sediment, (µg/kg or mg/kg)
- SWSL = Surface water screening level, (µg/L)
- SdSL = Sediment screening level, (µg/kg or mg/kg)

An EEQ for terrestrial plants and invertebrates will be calculated as follows:

$$EEQ = \frac{C_{ss}}{SSSL}$$

Where:
- EEQ = Ecological effects quotient (unitless)
- C_{ss} = Contaminant concentration in surface soil (µg/kg or mg/kg)
- SSSL = Plant or invertebrate screening level in soil (µg/kg or mg/kg)

The EEQ for the terrestrial wildlife model will be calculated as follows:

$$EEQ = \frac{Dose}{TRV}$$

Where: EEQ = Ecological effects quotient (unitless)
Dose = Daily intake dose (mg/kg-day)
TRV = Terrestrial reference value (NOAEL or LOAEL) (mg/kg-day)

D.6 ECOLOGICAL RISK UNCERTAINTY ANALYSIS

This section presents some of the uncertainties associated with ecological risk assessments.

D.6.1 Measurement and Assessment Endpoints

Measurement endpoints are used to evaluate the assessment endpoints that are selected for the SERA. For this SERA, the measures of effects are not the same as the assessment endpoints. Therefore, the measures are used to predict effects to the assessment endpoints by selecting surrogate species that will be evaluated. For example, a decrease in reproduction of a shrew is used to assess a decrease in reproduction of the small mammal population. However, predicting a decrease in reproduction of a shrew may either under- or overprotect the small mammal population, resulting from differences in ingestion rates, toxicity, food preferences, etc. between different species.

Risks to reptiles and amphibians will not be quantitatively evaluated because exposure factors are not established for most species, and toxicity data are very limited. However, risks to reptiles and amphibians will be qualitatively evaluated as part of the SERA.

D.6.2 Exposure Characterization

The contaminant dose to terrestrial wildlife is calculated using an equation that incorporates ingestion rates, body weights, bioaccumulation factors, and other exposure factors. These exposure factors are obtained from literature studies or predicted using various equations. Ingestion rates and body weights vary among species, especially among species inhabiting different areas. For example, food ingestion rates for the robin were reported as 0.89 gram/gram-day (g/g-day) in California and 1.52 g/g-day in Kansas (U.S. EPA, 1993b).

Bioaccumulation of contaminants into various biological media (i.e., plants, invertebrates, small mammals) depends on characteristics of the media such as pH, organic carbon, etc. Therefore, actual bioaccumulation factors at the site may be different than those used in the ERA that were obtained from the literature. Also, the bioavailability of the chemicals is not taken into account in this SERA. All the chemicals are assumed to be 100 percent bioavailable at the detected concentrations, which is unlikely to occur for contaminants in the environment.

There is uncertainty in the chemical data that are collected at the site. Measured levels of chemicals are only estimates of the true site chemical concentrations. For samples that are deliberately biased toward known or suspected high concentrations, predicted doses probably will be higher than actual doses.

Finally, under the conservative exposure scenario, terrestrial wildlife are assumed to live and feed only at the site. These assumptions will tend to overpredict risk because it is unlikely that most receptors will obtain all their food from within the site boundaries and from the most contaminated areas.

D.6.3 Ecological Effects Data

There is uncertainty in the ecological toxicity value comparison. The water-quality criteria developed by U.S. EPA in theory protects 95 percent of the exposed species. Therefore, some sensitive species may be present at the site that are not protected by the use of these criteria. There also are situations where the surface water screening levels (SWSLs) are over-predictive of risk if the sensitive species used to develop the criteria do not inhabit the site. Finally, with the exception of hardness for a few metals, the SWSLs do not account for site-specific factors, such as TOC or pH that may affect toxicity.

Potential adverse impacts to aquatic receptors from constituents in the sediment are evaluated by comparing the COPC concentration to sediment screening levels (SdSLs). The SdSLs have more uncertainty associated with them than do the SWSLs for the following reasons: The procedures for developing them are not as well established so screening levels have been developed using different methodologies and there are fewer sediment toxicity data than surface water toxicity data. Sediment characteristics (i.e., pH, acid volatile sulfides, total organic carbon) also may have a large impact on the bioavailability and toxicity of constituents.

Potentially adverse impacts to terrestrial plants and invertebrates from constituents in the surface soil are evaluated by comparing the COPC concentration to surface soil screening levels (SSSLs). The SSSLs are similar to the sediment screening levels in that they are less established than the SWSLs. Fewer studies and fewer data are available for establishing SSSLs than SdSLs and many of the SSSLs are

based on the results of only a few studies. In addition, the SSSLs are based on different endpoints, depending on the preference of the agency that developed them. Therefore, they have more uncertainty than surface water and sediment screening values.

The NOAELS and LOAELs that were selected for the wildlife endpoint species were based on species other than the endpoint species (i.e., rats, mice, ducks). There is uncertainty in the application of toxicity data across species because the contaminant may be more or less toxic to the endpoint species than it was to the test study species.

The toxicity of chemical mixtures is not well understood. All the toxicity information used in the ERA for evaluating risk to the ecological receptors is for individual chemicals. Chemical mixtures can affect the organisms very differently than the individual chemicals because of synergistic or antagonistic effects.

Finally, toxicological data for a few of the COPCs are limited or do not exist. Therefore, there is uncertainty in any conclusions involving the potential impacts to ecological receptors from these constituents.

D.6.4 Risk Characterization

Risks are possible if an EEQ is greater than or equal to unity regardless of the magnitude of the EEQ. However, the magnitude of effects to ecological receptors cannot be inferred based on the magnitude of the EEQ. Rather, an EEQ greater than 1.0 simply indicates that the dose used to derive the toxicity reference value was exceeded. Finally, there is uncertainty in how the predicted risks to a species at the site translate into risk to the population in the area as a whole.

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TABLE D-1
EXPOSURE PARAMETERS FOR TEST SPECIES AND SURROGATE WILDLIFE SPECIES
NAVAL SURFACE WARFARE CENTER, CRANE, INDIANA

Species	Body Weight (kg)			Food Ingestion Rate (kg/day)		Water Ingestion Rate (L/day)		Soil/Sediment Ingestion Rate (kg/day)		Home Range (acres) [6]		
	Avg.	Min.	Max.	Avg.	Conserv.	Avg.	Conserv.	Avg.	Conserv.	Avg.	Min.	Max.
Potential Test Species[1]												
Rat[2]	0.35	NA	NA	NA		NA		NA		NA		
Old field mouse[3]	0.014	NA	NA	NA		NA		NA		NA		
Mouse[2]	0.03	NA	NA	NA		NA		NA		NA		
Rabbit[2]	3.8	NA	NA	NA		NA		NA		NA		
Mink[5]	1	NA	NA	NA		NA		NA		NA		
Rhesus monkey[4]	5	NA	NA	NA		NA		NA		NA		
Beagle[2]	10	NA	NA	NA		NA		NA		NA		
Surrogate Wildlife Species [5]												
Bobwhite Quail	0.177	0.162	0.1855	0.0144	0.0164	0.0184	0.0231	0.001181	0.001345	28.6	15.8	41.3
American Robin	0.081	0.0773	0.0862	0.0976	0.1231	0.0113	0.0121	0.01015	0.012802	1.19	0.37	2
Short-Tailed Shrew	0.0169	0.0153	0.0192	0.0103	0.0162	0.0038	0.0043	0.001339	0.002106	0.97	0.97	0.97
Meadow Vole	0.0366	0.0329	0.0391	0.0119	0.0128	0.0064	0.0077	0.000286	0.000307	0.03	0.164	1.06
Raccoon	6.865	5.34	8.86	0.3347	0.4128	0.5664	0.5698	0.031462	0.038803	385.5	266.9	504.1
Belted Kingfisher	152	136	170	0.0689	0.0758	0.0167	0.0187	0.001378	0.001516	2.862	0.964	5.399

Notes:
See Attachment D.A-2-1 for the source of calculation of the exposure factors
NA - Not Applicable
1 - Only one value was provided so it was placed in the average column and some generic body weights may have been replaced by study-specific body weights
2 - USEPA, 1988
3 - Silva and Downing, 1996
4 - Barsotti et al, 1976
5 - USEPA, 1993b for all factors except soil ingestion; Beyer (1993) or Talmage and Walton (in press) for soil ingestion rates
6 - Home range for the kingfisher is presented in km of shoreline.
Avg - Average
Min - Minimum
Max - Maximum
Conserv - Conservative

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FIGURE D-1

NAVY'S ECOLOGICAL RISK ASSESSMENT TIERED APPROACH
NSWC CRANE, CRANE INDIANA

RPM Input and Risk Management Consideration[3]

Step 8: Risk Management

Tier 1. Screening Risk Assessment (SRA): Identify pathways and compare exposure point concentrations to bench marks.

Step 1: Site Visit; Pathway Identification/Problem Formulation; Toxicity Evaluation

Step 2: Exposure Estimate; Risk Calculation (SMDP)[1]

Proceed to Exit Criteria for SRA

Exit Criteria for the Screening Risk Assessment: Decision for exiting or continuing the ecological risk assessment.

1) Site passes screening risk assessment: A determination is made that the site poses acceptable risk and shall be closed out for ecological concerns.

2) Site fails screening risk assessment: The site must have both complete pathway and unacceptable risk. As a result the site will either have an interim cleanup or moves to the second tier.

Tier 2. Baseline Ecological Risk Assessment (BERA): Detailed assessment of exposure and hazard to "assessment endpoints" (ecological qualities to be protected). Develop site specific values that are protective of the environment.

Step 3a: Refinement of Conservative Exposure Assumptions[2] (SRA) - Proceed to Exit Criteria for Step 3a

Step 3b: Problem Formulation - Toxicity Evaluation; Assessment Endpoints; Conceptual Model; Risk Hypothesis (SMDP)

Step 4: Study Design/DQO - Lines of Evidence; Measurement Endpoints; Work Plan and Sampling & Analysis Plan (SMDP)

Step 5: Verification of Field Sampling Design (SMDP)

Step 6: Site Investigation and Data Analysis [SMDP]

Step 7: Risk Characterization

Proceed to Exit Criteria For BERA

Exit Criteria Step 3a Refinement

1) If re-evaluation of the conservative exposure assumptions (SRA) support an acceptable risk determination then the site exits the ecological risk assessment process.

2) If re-evaluation of the conservative exposure assumptions (SRA) do not support an acceptable risk determination then the site continues in the Baseline ecological Risk Assessment process. **Proceed to Step 3b.**

Exit Criteria Baseline Risk Assessment

1) If the site poses acceptable risk then no further evaluation and no remediation from an ecological perspective is warranted.

2) If the site poses unacceptable ecological risk and additional evaluation in the form of remedy development and evaluation is appropriate, proceed to third tier.

Tier 3. Evaluation of Remedial Alternative (RAGs C)

a. Develop site specific risk based cleanup values.

b. Qualitatively evaluate risk posed to the environment by implementation of each alternative (short term) impacts and estimate risk reduction provided by each (long-term) impacts; provide quantitative evaluation where appropriate. Weigh alternative using the remaining CERCLA 9 Evaluation Criteria. Plan for monitoring and site closeout.

Notes: 1) See EPA's 8 Steps ERA Process for requirements for each Scientific Management Decision Point (SMDP).
2) Refinement includes but is not limited to background, bioavailability, detection frequency, etc.
3) Risk Management is incorporated throughout the tiered approach.

FIGURE D-2

**ECOLOGICAL CONCEPTUAL SITE MODEL
JEEP TRAIL AND LITTLE SULPHUR CREEK
NSWC CRANE, CRANE, INDIANA**

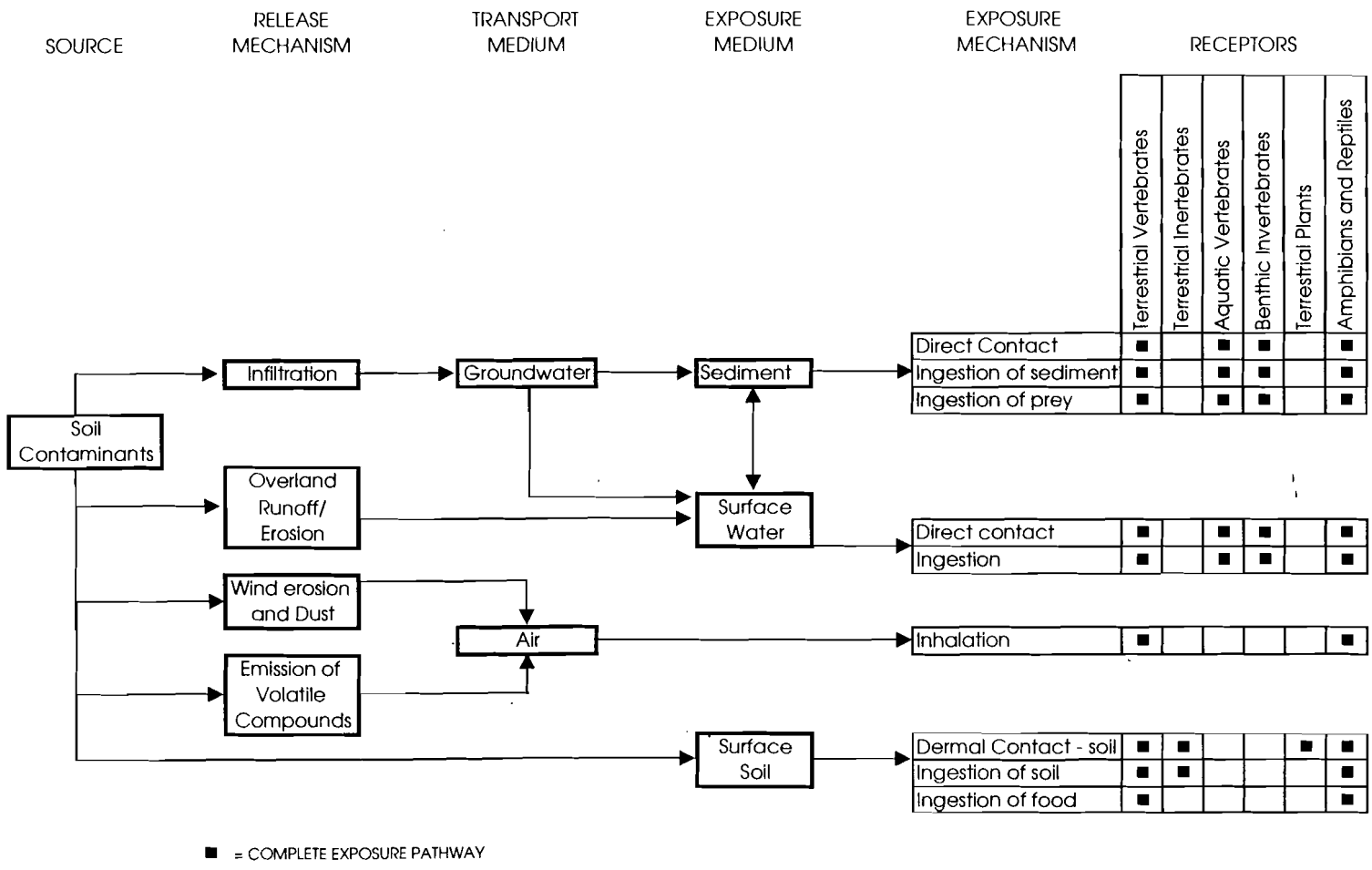
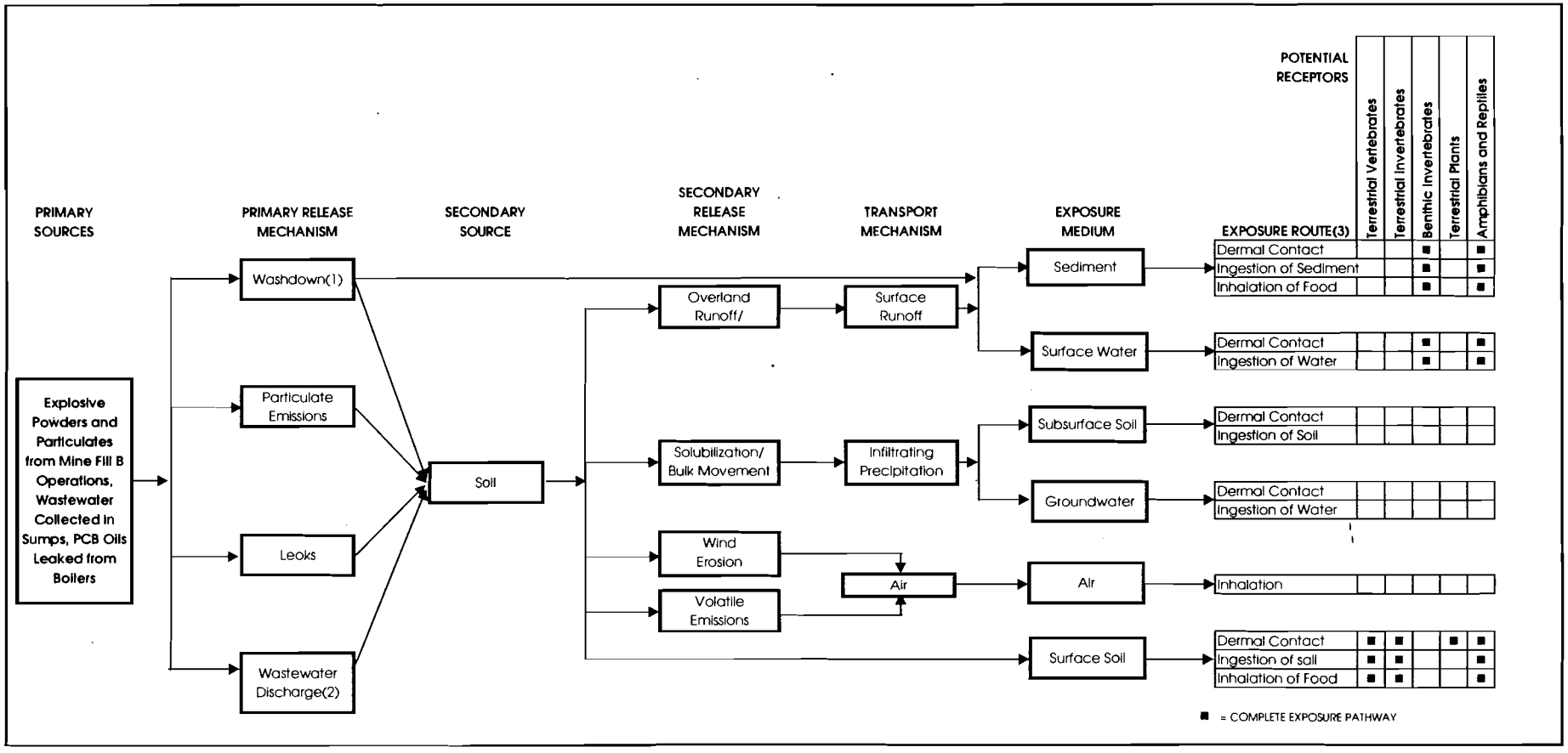


FIGURE D-3

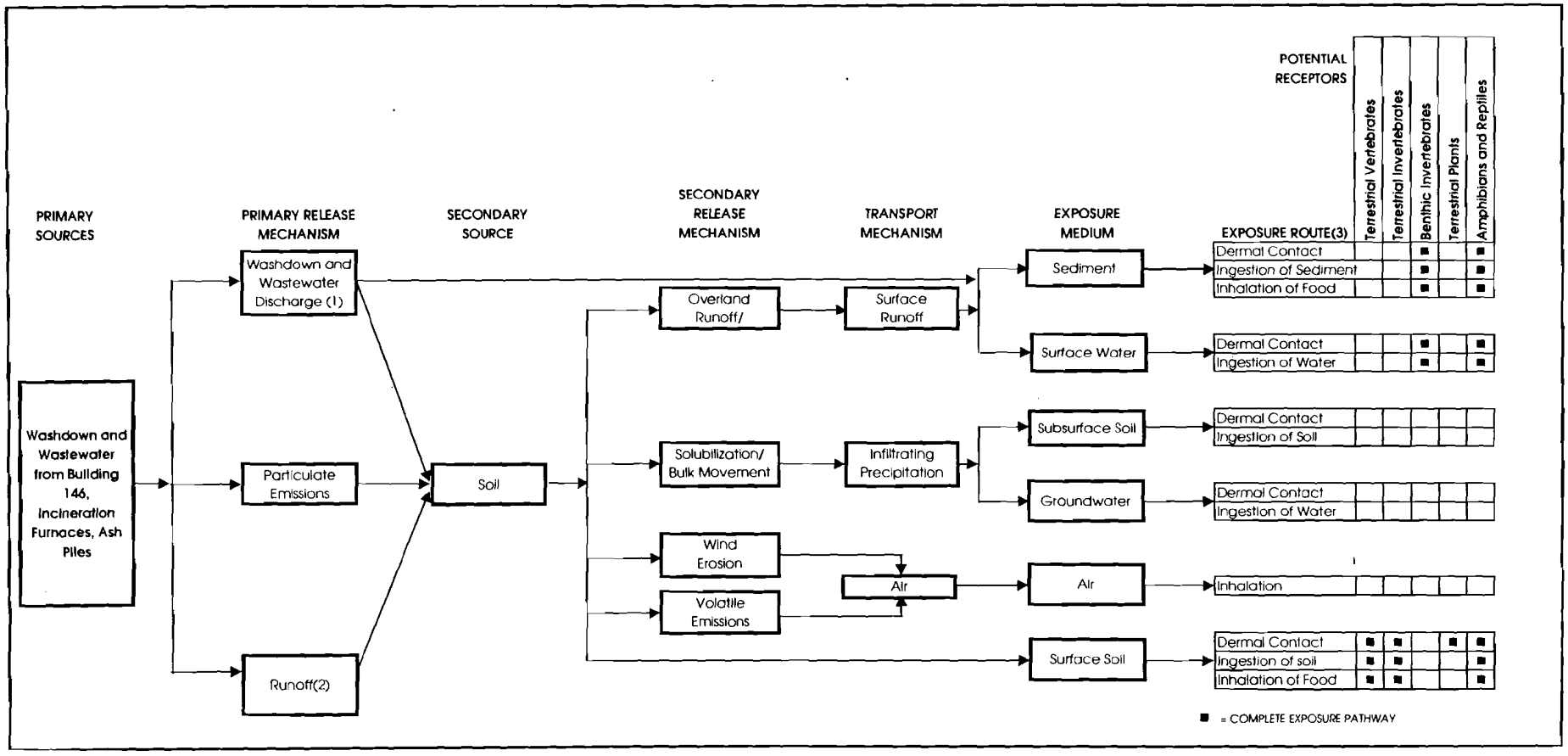
ECOLOGICAL CONCEPTUAL SITE MODEL
SWMU 13 - MINE FILL B
NSWC CRANE, INDIANA



1. Washdown of powders and particulates from rooftops.
2. Wastewater containing explosives was collected in sumps which released the wastewater directly to surface drainage which flows into Boggs Creek.
3. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

FIGURE D-4

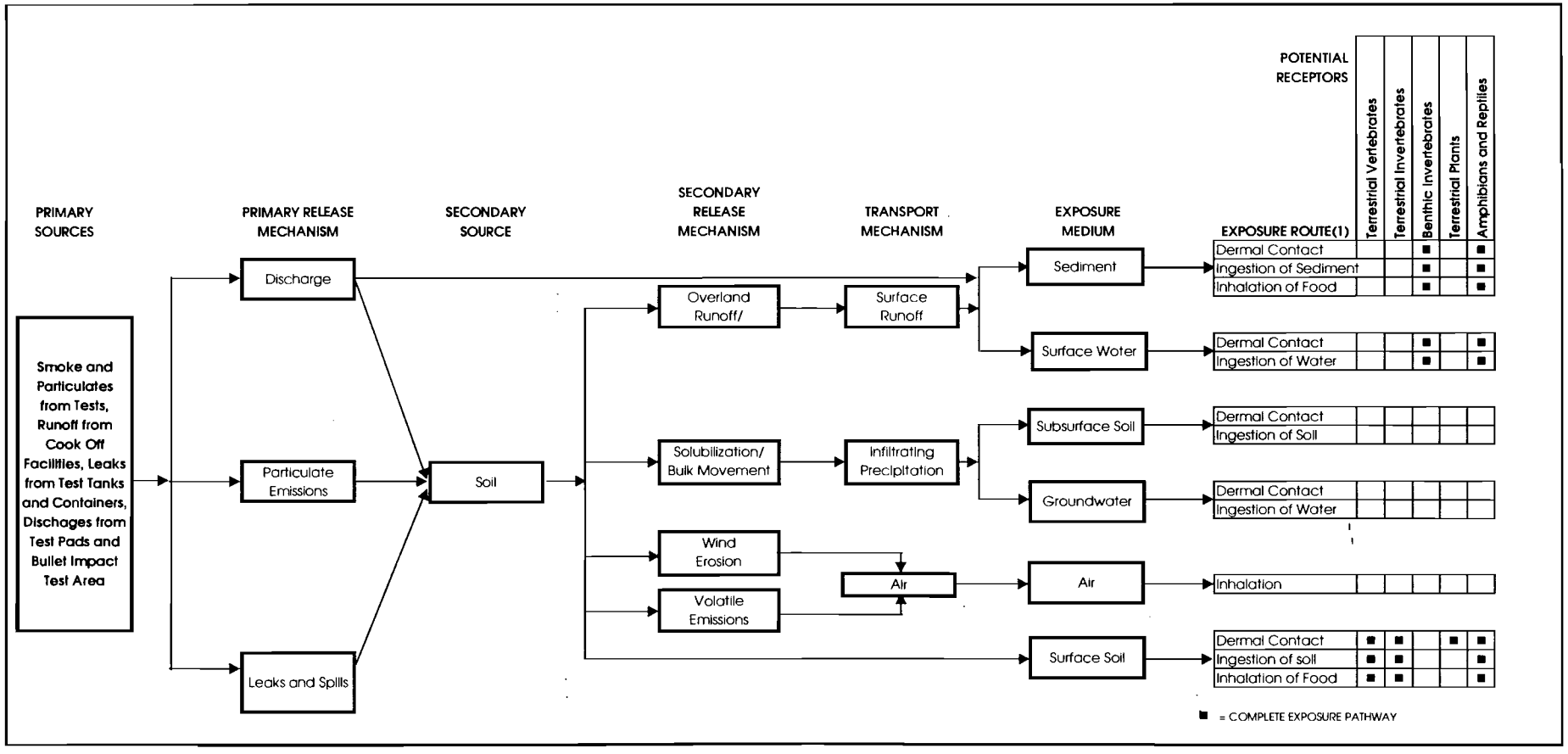
ECOLOGICAL CONCEPTUAL SITE MODEL
SWMU 16 - CAST HIGH EXPLOSIVE FILL / INCINERATOR BUILDING 146
NSWC CRANE, INDIANA



1. Washdown and washout wastewater were discharged through floor drains of Building 146 to sumps which emptied into the Boggs Creek watershed.
2. Runoff from incinerator ash piles.
3. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

FIGURE D-5

HUMAN HEALTH CONCEPTUAL SITE MODEL
SWMU 19 - PYROTECHNIC TEST AREAS
NSWC CRANE, INDIANA



1. Blank space indicates incomplete exposure pathway or relatively insignificant, or not applicable potential exposure.

ATTACHMENT D-1
RECEPTOR PROFILES
NSWC CRANE, CRANE, INDIANA

The following sections present the receptor profiles for the meadow vole, American robin, northern bobwhite quail, short-tailed shrew, raccoon, and belted kingfisher. The majority of the information for the profiles was obtained from the Wildlife Exposure Factors Handbook (U.S. EPA, 1993). The data for the incidental soil ingestion rates were obtained from the Estimates of Soil Ingestion by Wildlife (Beyer, 1993) except for the shrew data, which was in Talmage and Walton (undated). The exposure parameters that were selected for this SERA work plan are based on animals collected in or near southern Indiana, when available.

The food and water ingestion rate are presented in g/g (of body weight)-day on a wet weight basis. The home ranges for all species are presented in hectares in U.S. EPA (1993), but were converted to acres in this work plan by multiplying the number of hectares by 2.471. The only exception is the kingfisher's range, which is presented in km of shoreline. Also note that the estimated percent of soil in the diets are listed in dry weight, while the other exposure factors are in wet weight. The soil dry weight was not converted to a wet weight in this work plan because the percent moisture of the soils is not known. Also, incidental soil ingestion is only a small portion of the overall diet (2.0 to 13 percent).

The attached Table D.A-1 presents the calculation of the exposure parameters. Note that in Table D.A-1, the ingestion rates in kg/day (or L/day) for the conservative scenario was calculated by multiplying the maximum ingestion rate in g/g-day by the average body weight, while the ingestion rates in kg/day (or L/day) for the average scenario was calculated by multiplying the average ingestion rate in g/g-day by the average body weight. Typically, a minimum body weight is used in the conservative models. However, using the minimum body weight to calculate the maximum ingestion rate sometimes causes the conservative ingestion rate to be lower than the average ingestion rate. Therefore, the average body weight was selected to ensure that the ingestion rate for the conservative scenario was higher than the ingestion rate for the average scenario. The minimum body weight will be used in the dose equation for the conservative scenario. The only exceptions to this were for the food ingestion rate for the raccoon, and the water ingestion rates for the shrew, robin, and kingfisher. Because only one ingestion rate was available, the maximum body weights were used to calculate the conservative ingestion rates and the average body weights were used to calculate the average ingestion rates.

Meadow Vole (*Microtus pennsylvanicus*)

Meadow voles inhabit grassy fields, marshes, and bogs; however, they prefer fields with more grass, more cover, and fewer woody plants. They typically consume green succulent vegetation, sedges, seeds, roots, bark, fungi, insects, and animal matter. Green succulent vegetation makes up the majority of their diet.

The adult body weight for the vole in southern Indiana ranged from 0.0329 to 0.0391 kg with an average of 0.0366 kg. The only listed food ingestion rates were for voles in Russia, which ranged from 0.30 to 0.35 g/g-day, with an average of 0.325 g/g-day. The water ingestion rates are 0.14 (estimated) and 0.21 g/g-day, with an average of 0.175 g/g-day. Finally, the incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (2.4 percent) from Beyer (1993).

The home range for the meadow vole was calculated using data from a Michigan old field. The values ranged from 0.0297 to 1.06 acres with an average home range of 0.164 acres.

American Robin (*Turdus migratorius*)

American robins inhabit parks, lawns, moist forest, swamps, open woodland, and orchards. Robins forage on the ground in open areas, along habitat edges, or the edges of streams. They also may forage above ground in shrubs and within the lower branches of trees. In the months proceeding and during the breeding season, robins feed primarily on invertebrates and on some fruits. During the rest of the year their diet consists primarily of fruits.

The adult body weight for the American robin in New York woodlands and forests, and in Pennsylvania ranged from 0.0773 to 0.0862 kg with an average of 0.081 kg. The only listed food ingestion rates were for robins in Kansas (1.52 g/g-day) and California (0.89 g/g-day), with an average of 1.205 g/g-day. The water ingestion rate was estimated as 0.14 g/g-day. The incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (10.4 percent for a woodcock) from Beyer (1993).

The home range for the robin was calculated using data from an Ontario forest. The values ranged from 0.37 to 2 acres with an average home range of 1.19 acres.

Northern Bobwhite Quail *(Colinus virginianus)*

Quails inhabit grasslands, idle fields, pastures, and large clumps of grasses. Bobwhites forage in areas with open vegetation, some bare ground, and light litter. Seeds from weeds, woody plants, and grasses comprise the majority of an adult's diet, although green vegetation has been found to dominate their diet in winter in the south.

The adult body weight for the bobwhite quail in southern Illinois agricultural areas ranged from 0.162 to 0.1855 kg with an average of 0.177 kg. The listed food ingestion rates are for quails in Kansas and Texas lab studies, and in a captive study from Massachusetts. The values ranged from 0.067 to 0.093 g/g-day, with an average of 0.082 g/g-day. The water ingestion rate was estimated as 0.10 and 0.11 g/g-day, and measured in a Texas lab (0.86 to 0.131 g/g-day), for an average water ingestion rate of 0.104 g/g-day. The incidental soil ingestion rate is calculated by multiplying the food ingestion rate by the percentage of soil that is incidentally ingested (0.082 percent for a Canada goose) from Beyer (1993).

The home range for the quail was calculated using data from southern Illinois idle farms, woods, brush, and cornfields. The values for individuals ranged from 15.8 to 41.3 acres with an average home range of 28.6 acres.

Short-Tailed Shrew (*Blarina brevicauda*)

Shrews inhabit a wide variety of habitats and are common in areas with abundant vegetative cover. They need cool, moist habitats because of their high metabolic and water-loss rates. The short-tailed shrew is primarily carnivorous, eating insects such as earthworms, slugs, and snails.

The adult body weigh for the short-tailed shrew in various Pennsylvania habitats ranged from 0.01525 to 0.01921 kg with an average of 0.01687 kg. The listed food ingestion rates are for shrews in labs in Ohio (0.49 g/g-day), Wisconsin (0.43 to 0.96 g/g-day), and Virginia (0.541 g/g-day), with an average of 0.61 g/g-day. The water ingestion rate was determined as 0.223 g/g-day in an Illinois laboratory. The incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (13 percent) from Talmage and Walton (undated).

The home range for the shrew was calculated using data from a tamarak bog in Manitoba (only value available). The value was 0.9699 acres.

Raccoon (*Procyon lotor*)

Raccoons are found near virtually every aquatic habitat, particularly in hardwood swamps, mangroves, floodplain forests, and freshwater and saltwater marshes. They are also common in suburban residential areas. They use surface waters for both drinking and foraging. They feed primarily on fleshy fruits, nuts, acorns, and corn, but also eat grains, insects, frogs, crayfish, eggs, and virtually any animal and vegetable matter.

The adult body weights based on data from Illinois, range from 5.34 to 8.86 kg, with an average of 6.865 kg. The average food ingestion rate of 0.3347 kg/day was calculated using the average body weight and the following equation from U.S. EPA (1993):

$$FI = (0.0687)\ (BW^{0.822})$$

Where: FI = Food ingestion rate (kg/day)
BW = Body weight in kg

The range of water ingestion rates is listed as 0.082 to 0.083 g/g-day. The incidental soil ingestion rate is calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingestion (0.094), as presented in Beyer (1993).

Based on data from Michigan, home range sizes for the raccoon range from 266.9 to 504.1 acres for an average home range of 385.5 acres.

Belted Kingfisher (*Ceryle alcyon*)

Belted kingfishers are typically found along rivers, streams, and the edges of lakes and ponds. They are also common along seacoasts and estuaries. They prefer water that is free of thick vegetation and overhanging trees that obscure the view of the water. Because kingfishers eat primarily fish that swim near the surface or in shallow water, they require relatively clear water to see and catch their prey. Although kingfishers feed predominantly on fish, they have been known to consume crayfish, crabs, mussels, lizards, frogs, toads, small snakes, turtles, insects, salamanders, newts, young birds, mice, and berries.

Based on data from Michigan, Pennsylvania, Ohio, and Minnesota, the adult body weights range from 0.136 to 0.170 kg, with an average of 0.152 kg. The listed food ingestion rates, based on data from Michigan, range from 0.41 to 0.5 g/g-day. The water ingestion rate is estimated as 0.11 g/g-day. The

incidental soil ingestion rate was calculated by multiplying the ingestion rate by the percentage of soil that is incidentally ingested (2 percent), based on the mallard data presented in Beyer (1993).

The home range for the kingfisher ranges from 0.39 to 2.185 km of shoreline, based on data from streams in Pennsylvania and Ohio.

REFERENCES

Beyer, N., E. Connor, and S. Gerould. 1993. Estimates of Soil Ingestion by Wildlife. Patuxent Wildlife Research Center, Laurel, MD.

EPA, 1993. Wildlife Exposure Factors Handbook. U.S. Environmental Projection Agency. Office of Research and Development. Washington, D.C. December 1993. EPA/600/R-93/187a.

Sample B.E., M.S. Aplin, R.A. Efroymson, G.W., Suter II, and C.J.E. Welsh. 1997. Methods and Tools for Estimation of the Exposure of Terrestrial Wildlife to Contaminants. Oak Ridge National Laboratory. October. ORNL/TM-13391.

Talmage, Sylvia S., and Barbara T. Walton. In Press. "Food Chain Transfer and Potential Renal Toxicity of Mercury to Small Mammals at a Contaminated Terrestrial Field Site." Health and Safety Research Division and Environmental Science Division, Oak Ridge National Laboratory.

TABLE D.A-1

CALCULATION OF EXPOSURE PARAMETERS FOR WILDLIFE RECEPTORS
NAVAL SURFACE WARFARE CENTER, CRANE, INDIANA

Exposure Parameters	Meadow Vole		Short-Tailed Shrew		American Robin	Bobwhite Quail		Raccoon		Belted Kingfisher
Body Weights (g)	32.9	35.5	17.61	16.87	77.3	180	181	7090	6160	150
	39.1	39	17.33	15.58	80.8	168	183	7140	6440	136
			19.21	15.7	86.2	162	179	7600	5340	158
			17.4	15.25	83.6	175	175	6000	5620	147
					77.4	178	183.2	6400	8860	148
					80.6	179	185.5	7740	7560	170
						180	173	6560	7600	
						162.8	180.4			
Minimum	***32.9***		***15.25***		***77.3***	***162***		***5340***		***136***
Maximum	***39.1***		***19.21***		***86.2***	***185.5***		***8860.0***		***170.0***
Average	***36.63***		***16.87***		***81.0***	***177***		***6865***		***152***
Food Ingestion Rate (g/g-day) [1]	0.3	0.35	0.49	0.77	0.89	0.067	0.079			0.5
			0.62	0.55	1.52	0.072	0.093			0.41
			0.43	0.96		0.09	0.089			
			0.52	0.54						
Minimum	***0.3***		***0.43***		***0.89***	***0.067***				***0.41***
Maximum	***0.35***		***0.96***		***1.52***	***0.093***				***0.5***
Average	***0.325***		***0.61***		***1.205***	***0.082***				***0.455***
Food Ingestion Rate (kg/day)										
Conservative	***0.0128***		***0.0162***		***0.1231***	***0.0164***		***0.4128***		***0.0758***
Average	***0.0119***		***0.0103***		***0.0976***	***0.0144***		***0.3347***		***0.0689***
Water Ingestion Rate (g/g-day)	0.14	0.21	0.223		0.14	0.115	0.1	0.082	0.083	0.11
						0.106	0.131			
						0.093	0.101			
						0.086	0.102			
						0.11	0.1			
Minimum	***0.14***		***0.223***		***0.14***	***0.086***		***0.082***		***0.11***
Maximum	***0.21***		***0.223***		***0.14***	***0.131***		***0.083***		***0.11***
Average	***0.175***		***0.223***		***0.14***	***0.104***		***0.083***		***0.110***
Water Ingestion Rate (L/day)										
Conservative	***0.0077***		***0.0043***		***0.0121***	***0.0231***		***0.5698***		***0.0187***
Average	***0.0064***		***0.0038***		***0.0113***	***0.0184***		***0.5664***		***0.0167***
Home Range (Ha) [2]	0.43	0.097	0.3925		0.15	7.6	6.4	204	108	2.185
	0.019	0.041			0.81	16.7	15.6			1.028
	0.013	0.033								1.03
	0.012	0.013								0.39
	0.043	0.057								
	0.023	0.032								
	0.051	0.078								
	0.058	0.061								
Minimum (acres) [2]	***0.0297***		***0.9699***		***0.37***	***15.8***		***266.9***		***0.39***
Maximum (acres) [2]	***1.06***		***0.97***		***2.00***	***41.3***		***504.1***		***2.19***
Average (acres) [2]	***0.164***		***0.970***		***1.19***	***28.6***		***385.5***		***1.16***

Notes:
Source of data is U.S. EPA, 1993
1 - Food Ingestion Rate (kg/day) for the raccoon was calculated using the following equation:
$FI = (0.0687)(BW^{0.822})$, where FI = Food Ingestion Rate (kg/day) and BW = Body Weight (kg).
2 - Home range for the kingfisher is presented in km of shoreline.
Ingestion Rates (kg/day or L/day) (if more than 1 ingestion rate is available)
- Conservative value = Max Ingestion Rate (g/g-day)* Avg. Body Weight
- Average value = Avg. Ingestion Rate (g/g-day)*Avg. Body Weight
Ingestion Rates (L/day) (if only 1 ingestion rate is available)
- Conservative value = Ingestion Rate (g/g-day)* Max. Body Weight
- Average value = Ingestion Rate (g/g-day)*Avg. Body Weight

APPENDIX E

EXAMPLE WILCOXON RANK-SUM TEST

CLIENT NSWC CRANE	JOB NUMBER CTO166 N3994.0000.JE004013 5
SUBJECT SAMPLE CALCULATIONS OF STATISTICAL ANALYSIS	
BASED ON	DRAWING NUMBER
BY MBP / CHECKED BY BEL	APPROVED BY / DATE 6/3/99

USING ALUMINUM DATA FOR SUBSURFACE SOIL SAMPLES AT SITE 11, CALCULATION OF Z_{rs} VALUE IS PERFORMED VIA WILCOXON RANK-SUM TEST

① CALCULATION OF SUM OF RANKS (W_{rs}) [mg/kg]

SITE DATA	SAMPLE	BKGND DATA	SAMPLE	SITE RANKS	BKG RANK
9430	11-SB01-1416	21800	BG-04-SB	10	15
5080	11-LW-03	10600	BG-05-SB	5	12
2860	11-SB05-1416	5780	BG-06-SB	2	7
3880	11-LW-06	22000	BG-07-SB(6)	4	16
10800	11-SB07-0607	40700	BG-10-SB(6)	13	18
7740	11-SB08-1416	9900	BG-12-SB(3)	8	11
5310	11-SB09-1416	22900	BG-13-SB(6)	6	17
2940	11-SB10-1012	20000	BG-SB-100(7)	3	14
1340	11-SB11-1214	8100	BG-SB-102(7)	1	9
				$\Sigma = 52$	$\Sigma = 119$

$^{*}W_{rs}$ (SITE) = 52

② CALCULATION OF Z_{rs}

$$Z_{rs} = \frac{W_{rs} - \frac{n_1(m+1)}{2}}{\sqrt{\frac{n_1 n_2 (m+1)}{12}}}$$

Where: W_{rs} = SUM OF RANKS (SITE) n_1 = No. SAMPLES (SITE)

$m = n_1 + n_2$ n_2 = No. SAMPLES (BCKGD)

CLIENT NSWC CRANE	JOB NUMBER CTO166 N3994.0000.JE0040135
SUBJECT SAMPLE CALCULATIONS OF STATISTICAL ANALYSIS	
BASED ON	DRAWING NUMBER
BY MBP / CHECKED BY BEL	APPROVED BY / DATE 6/3/99

$$Z_{rs} = \frac{52 - \frac{9(18+1)}{2}}{\sqrt{\frac{81(18+1)}{12}}}$$

$$Z_{rs} = \frac{52 - 85.5}{\sqrt{128.25}}$$

$$Z_{rs} = \frac{-33.5}{11.32} = -2.959$$

$Z_{rs(CRITICAL)\,\alpha=.20} = 0.845$

SINCE $Z_{rs} = -2.959 < 0.845 = Z_{rs(critical)\alpha=.20}$, WE ACCEPT THE NULL HYPOTHESIS THAT THE SITE AND BACKGROUND SAMPLES WERE OBTAINED FROM THE SAME STATISTICAL POPULATION.

APPENDIX F

DATA MANAGEMENT PLAN

APPENDIX F
DATA MANAGEMENT PLAN

1.0 PROJECT PLANNING

A large amount of environmental and physical data has been collected in support of the Installation Restoration (IR) program. TtNUS has the responsibility of managing these data in a basewide relational database and Geographical Information System GIS. The contents of the database will be outlined in the Sitewide Data Catalog (which at a minimum, contains the data fields identified in Attachment F1 of this appendix). The data catalog will outline what data are contained within the database (by investigation, media, etc.), the generator of the data (TtNUS, Corps of Engineers, etc.), and the level of quality of the datum, where applicable. The catalog will note whether or not the analytical data were validated and to what level. It is the responsibility of the TtNUS data manager to coordinate with the NSWC Crane project team in order to keep the data catalog current and make the most recent version available to all team members. A copy of the data catalog will be maintained in the project central file at the office of TtNUS. It is the responsibility of the all team members to ensure that the data catalog is correct and current and to notify the TtNUS data manager of any newly generated data that will support the needs of the project.

Before every data collection event, the task order manager (TOM) will hold a kick-off meeting to outline the data needs of the task order and review the data flow process (Attachment F2). Attendees of the kick-off meeting should include the TOM, the human health risk assessment (HHRA) lead, the ecological risk assessment (ERA) lead, the field operations leader (FOL), the project chemist, the data management lead, and the GIS lead. The data management lead will distribute a copy of the database checklist (Attachment F3) and review the contents with the project team. The database checklist will allow the project team to determine how the data will be managed and manipulated in order to achieve the project needs and objectives. A completed copy of the database checklist will be maintained in the project central file and distributed to all members of the project team within 7 days of the kick-off meeting.

2.0 NEWLY GENERATED DATA

Upon a directive from SOUTHDIV to collect additional site data, the TOM will coordinate with the designated data management lead and GIS lead for the project. It is the responsibility of the FOL to comply with the sample and location nomenclature outlined in the QAPP. It is also the responsibility of the FOL to coordinate with the GIS lead to ensure that all survey technical specifications require the proper coordinate system, which is Indiana State Planar - North American Datum 1983 for the horizontal coordinates and National Geodetic Vertical Datum 1988 for the vertical coordinates.

Prior to field mobilization, the FOL will coordinate with the sample management coordinator (SMC) to initiate a sample tracking process. It is the responsibility of the TOM to ensure that a sampling tracking procedure is implemented. Sample Tracking Request Forms, a sample tracking database example, and example jar labels are included as Attachments F4, F5, and F6, respectively. In the event that a field change takes place, the FOL is required to complete the Field Task Modification Request (FTMR), which will be forwarded to all members of the project team.

According to all laboratory technical specifications for NSWC Crane, the analytical laboratories will be contractually required to deliver the analytical data in NSWC Crane standard Electronic Data Deliverable (EDD) format (Attachment F7). Particular attention should be paid to the EDD requirements for validated vs. non-validated data. When all samples and analyses have been accounted for, the SMC will forward the analytical data to TtNUS for incorporation into the NSWC Crane database, which is located on the Local Area Network (LAN) in Pittsburgh, Pennsylvania. The NSWC Crane database structure is presented in Attachment F8.

3.0 HISTORICAL DATA

In the event that the NSWC Crane project team decides that existing hard-copy data not outlined in the data catalog (Attachment F1) must be incorporated into the project database, SOUTHDIV will direct the appropriate consultant to incorporate the data into the project database. The data management lead will review the hard-copy data and prepare a summary of the samples and analyses that need to be entered. The format of the summary table should be similar to the sample tracking database provided in Attachment F5. It is the responsibility of the TOM to review the sample summary table and verify that the entry of these data will satisfy the project requirements. The data management lead will physically edit the hard-copy analytical data to clearly designate which information on the hard-copy must be entered into the database. Copies of the marked-up data must be distributed to two separate parties for entry into an Excel spreadsheet. Upon completion of the dual-key entry, the data management lead will electronically compare the two data files to identify discrepancies and correct the data appropriately. The database should then be queried against the sample summary table to ensure that all pertinent data have been entered and checked for accuracy.

The data management lead will coordinate with the GIS lead to acquire the sample location data (Attachment F8) for those samples that need to be entered. Sample location maps should be used to digitize the sample locations, using the base mapping layer in the GIS. To the extent possible, the GIS lead will capture, as metadata, the accuracy of the sample location maps used to digitize the location

coordinates. If no sample location maps or other positional information exist for the historical data, the project team should evaluate the utility of these data in the NSWC Crane database.

4.0 MAPPING AND GRAPHICS

CADD mapping is generally provided by the activity. TtNUS currently does not use metadata to track changes to the mapping. In addition, Tri-Service Spatial Data Standards (TSSDS) are not utilized unless the mapping from the base already incorporates them. TSSDS is not used in the final GIS because limited utility is gained from the substantial time required to incorporate the standards.

In addition to CADD mapping, Digital Ortho Quarter (DOQ) Quads, aerial photography, and United States Geological Survey 7.5 minute quads are obtained. The quads are obtained from either the USGS or other suppliers, and the aerial photography is provided by the activity. As necessary, the images are warped to the predetermined coordinate system using Microstation. Again, metadata are not used to track the changes. From survey data, sampling locations are organized, and then a sample-vs-location table is built so that the data can be loaded into the sample_data.dbf table (Attachment F8).

5.0 THE ENVIRONMENTAL GEOGRAPHIC INFORMATION SYSTEM (EGIS)

All environmental data collected in support of the NSWC Crane project will be incorporated into the GIS. The themes, layers, and database information contained in the GIS are outlined in the data catalog (Attachment F1). The NSWC Crane GIS will be made available to all members of the project team. CD-ROM EGIS deliverables will be made available upon request from SOUTHDIV.

6.0 ASSIMILATION OF DATA FROM OUTSIDE SOURCES

When environmental data are collected by a contractor other than TtNUS, it is the responsibility of the SOUTHDIV remedial project manager (RPM) to notify the TtNUS TOM. The RPM should forward a scope of work directing TtNUS to coordinate with the contractor and incorporate their data into the basewide GIS. To the extent possible, the RPM should direct the Navy contractor to supply the data to TtNUS in the format outlined in Attachment F8. When TtNUS has incorporated the data into the GIS, a hard-copy report will be sent to the contractor for verification that all pertinent data have been incorporated in a complete and accurate fashion.

7.0 SOFTWARE

TtNUS will standardize on the following software packages when managing and manipulating data for the NSWC Crane project:

- Data Management - Microsoft Visual FoxPro 6.0 (SQL Server 7.0)
- GIS - ArcView 3.2 (see Attachment F9 for instructions)
- 3-D Visualization - EVS Pro 5.11
- Ground Water Modeling - GMS 3.1
- Statistical Analysis - Statistica 5.5
- Terrain Analysis - TerraModel 9.7

8.0 STORAGE OF DATA

TtNUS utilizes Microsoft NT for Networks as its Information Management System (IMS). The NT IMS currently serves over 110 desktop computers. The NT IMS automatically backs up the system on a daily basis, thereby disallowing more than 1 day of work being lost should the network crash or malfunction. The database management and GIS groups have been allocated distinct drives on the LAN. All environmental data for the NSWC Crane Project will be stored in the \\nusrpitbdc1\sdiv\NSWC_Crane subdirectory of this drive on the NT Server. All tables, queries, programs, and reports will be saved in the NSWC_Crane.pjx file in Microsoft Visual FoxPro. The NSWC Crane EGIS will be stored in the \\nusrpitbdc1\gis\NSWC_Crane directory on the NT Server. All ArcView project files (*.apr) will be documented in a text file called readme_project.txt. This text file will also be stored in the \\nusrpitbdc1\gis\NSWC_Crane directory.

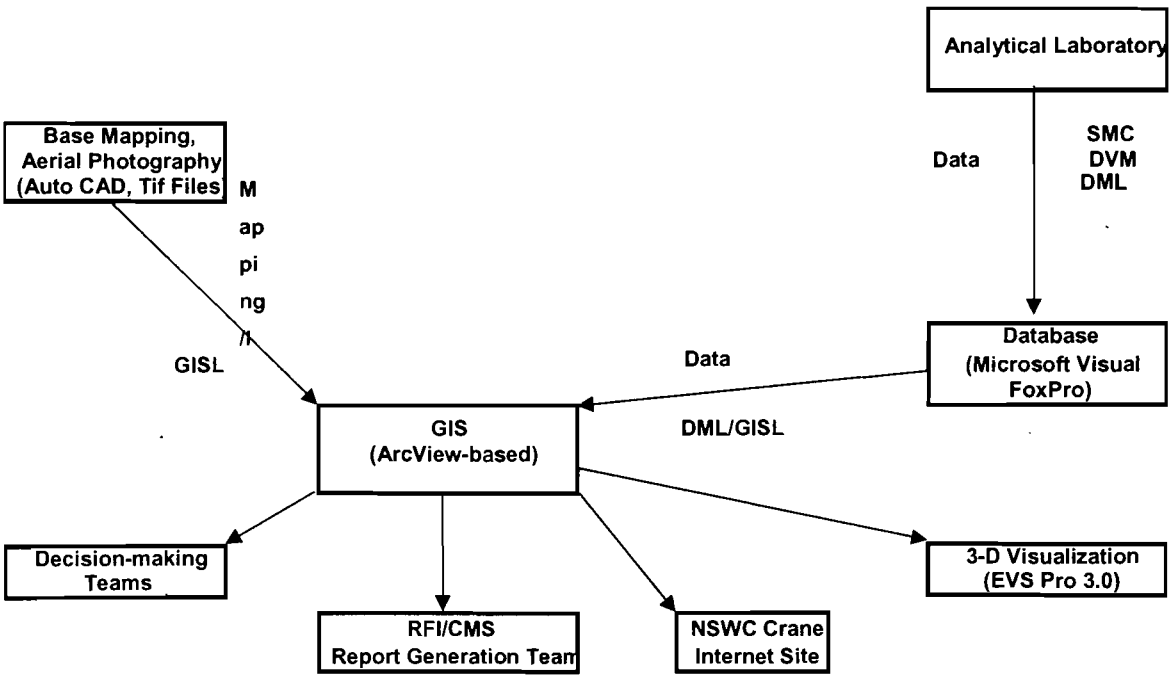
ATTACHMENT F1

DATA CATALOG
(Minimum Requirements)

Category	RFI Phase	Medium Sampled	Sample Type	No. of Samples	Fraction Analyzed

ATTACHMENT F2

THE DATA FLOW PROCESS



Notes:

SMC = Sample Management Coordinator.
DVM = Data Validation Manager.
DML = Data Management Leader.
GISL = GIS Leader.

ATTACHMENT F3

DATABASE CHECKLIST

DATABASE PLANNING CHECKLIST

PROJECT NAME: NSWC CRANE **PROJECT NUMBER: N3994**

TASK ORDER MANAGER: R. BASINSKI **PLANNING DATE: 7/05/01 TO 9/5/01**

1. Provide a general description of the project (regulatory authority, media to be sampled, approximate number of samples by media, analyses by media, data evaluation tasks required):
RCRA. Develop QAPP for all media Phase III RFI of SWMUs 12, 13, 16, and 19. No. of samples varies from SWMU to SMWU. Expect about 40 soil samples for SWMUs 16 (surface and subsurface) and 19 (almost all surface) and few soil samples for SMWUs 12 and 13. Expect about 15 ground water samples for each SWMU and 8 surface water samples. Expect about 8 sediment samples for each SWMU. QC samples are in addition to these estimates.

2. Provide a general description of the sample nomenclature that will be used for samples collected by Tetra Tech NUS:
Same CTOs 126/159 and 131/158 (SOP CTO131-7).

3. Will historical data be entered in the database? Yes No

4. Will historical data be used to define the nature and extent of contamination? Yes No
At least for SWMUs 12 and 13, and possibly SWMU 16.

5. Will historical data be used for risk assessment purposes? Yes No
At least for SWMUs 12 and 13, and possibly SWMU 16.

6. How much historical data exists (i.e., number of samples by matrix, analysis by matrix)?
As provided by NSWC Crane (several floppy disks). Amount of data is different for each SWMU. No data are available for SWMU 19.

7. In what format will the historical data be provided? Hardcopy Electronic

8. If historical data are in electronic form, what software was used and what is the format? MS Excel

9. If historical data are in hardcopy form, will Form I's, summary tables, or reports be provided? Copies of historical data will be necessary to generate a budget estimate.
Environmental Monitoring Reports, Interim Measures Reports are the sources of the hard copy data. No Laboratory data packages are available. Significant amounts of data were provided via e-mail and floppy disks.

10. Will Quality Assurance review of historical data be necessary? Yes No

11. If Quality Assurance review of historical data is necessary, describe the scope of the Quality Assurance review:

Data were already evaluated by EPA Region 5 and judged to be of adequate quality for this project. It is not known whether all data have been validated but it is assumed that they have been. EPA Region 5 has approved the use of the data for decision making.

12. Will a GIS database be necessary for the project. (Yes) No
If so, the GIS Group should be consulted for a budget estimate.

13. What nomenclature has been (will be) used to identify field duplicate samples?

Same as CTOs 126/159 and 131/158.

14. Will field duplicate results be averaged and presented as one result in the database? Will they be presented as distinct results, or will both the average and the distinct results be presented?

No averaging. Only the original result will be used to represent site concentrations. Duplicate results will be used only for evaluating data variability. Non-detects will be identified with "U" validation flag. When appropriate, half the MDL will be used in place of non-detect results when conducting statistical analyses.

15. How will the average value for duplicate samples be determined on a matrix-specific basis?

Not applicable

16. Are any unvalidated data to be included in the database? **See Item 11.** Yes (No)

17. Will unvalidated data be used for defining the nature and extent of contamination? Yes (No)

18. Will unvalidated data be used for risk assessment purposes? Yes (No)

19. Are any field screening (e.g., no-fixed base laboratory) data to be included in the database? Yes (No)

20. Will field screening data be used for defining the nature and extent of contamination? Yes (No)

21. Will field screening data be used for risk assessment purposes? Yes (No)

22. Will statistical correlation of laboratory and field screening data be necessary? Yes (No)

23. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to define nature and extent? Yes (No)

24. If a correlation exists between field screening and laboratory data, will the results of regression analysis be used to support the risk assessment? Yes (No)

25. Will fixed base laboratory field parameters be included in the database (e.g., pH, conductance, temperature)? Yes No

26. Will statistical correlations be necessary for TCLP versus total analysis data? Yes No

27. Will statistical correlations be necessary for filtered versus unfiltered samples? Yes No

28. Will any other statistical correlations be necessary? Yes No

29. Are there wells that have been screened in different aquifers? **Possibly** Yes No

30. Will data for various aquifers be segregated by depth? **Possibly** Yes No

31. Can the sample nomenclature system be used to identify wells in different aquifers? Yes No

32. Will samples from other matrices (soil, sediment, or surface water) be segregated by depth? Yes No

33. Can the sample nomenclature system be used to identify depth-specificity? Yes No

34. Have any removal actions been performed at the site? Yes No

If removal actions have been performed, plan and cross-sectional views reflecting the extent of the removal action must be provided. **Not applicable, as removed soil was treated and replaced.**

35. Will any composite sample results be included in the database? Yes No

36. If composite samples are included, how will they be used for the nature and extent of contamination?
Not applicable

37 If composite samples are included, how will they be used for the risk assessment?
Not applicable

38. Will the site be segregated into Areas of Concern, Solid Waste Management Units, etc? Yes No

39. Is the sample nomenclature adequate for such segregation? Yes No

If the sample nomenclature is inadequate for assigning samples to an AOC or SWMU, the Project Manager or designee must provide a base map or tabular summary clearly delineating the relationship between each sample and each AOC/SWMU.

40. Were any temporal samples collected (e.g., quarterly sampling of wells)? Yes No

41. If temporal samples were collected, how will they be used to define the nature and extent of contamination?
Not applicable

42. If temporal samples were collected, how will they be used to support the risk assessment?
Not applicable

43. Are State, Federal, or Regional criteria to be included in data summary tables? Yes No

44. Identify the criteria that must be presented in the summary tables.
Risk-based target levels, and screening levels, and background concentrations, as provided in the QAPP. These will be updated at the time of data evaluation.

45. Will State, Federal, or Regional criteria be used to select COPCs? Yes No

46. Identify the criteria to be used as COPC selection tools.
Risk-based target levels as provided in the QAPP and updated at the time of data evaluation.

47. Are filtered and unfiltered surface water samples differentiated? Yes No

48. If such samples are differentiated, how?
Identified as "filtered" or "unfiltered" or as "dissolved" or "total." Filtered ground water samples will be collected if the turbidity can not be reduced to less than 10 NTUs.

49. Which of these samples will be used for the human health risk assessment?
Surface Water Filtered Unfiltered
Groundwater Filtered Unfiltered

50. Which of these samples will be used for the ecological assessment?
Surface Water Filtered Unfiltered
Groundwater Filtered Unfiltered

51 Will background data be included in the database? Soil and water Yes No

52. How are background samples identified?
As described in the QAPP.

53. Will background results be used to support selection of COPCs? Yes No

54. What statistical analyses will be required for the background data?
As described in the QAPP Sections 1 and 8 (maximum values and data set comparison, etc.)

55. Will background data be segregated by depth? Yes No

56. What background matrices must be segregated by depth?
Soils and ground water. Will segregate soils as done for the NSWC Crane Basewide Soil Background Investigation Report, 2001, TtNUS. Ground water will be segregated according to screen depth of upgradient versus downgradient wells.

57. What format will be used for data presentation (e.g., appendices and summary tables, comprehensive text tables, tag maps, isoconcentration contours, etc.)?
All of the above and perhaps more, depending on the outcome of the investigation.

ATTACHMENT F4

SAMPLE TRACKING REQUEST FORM

SAMPLE TRACKING AND DATA MANAGEMENT AT PROJECT INCEPTION

PROJECT START-UP CHECKLIST

ATTACHED IS A PROJECT START-UP CHECKLIST (CAN BE FOUND IN DATA MANAGEMENT CENTRAL FILE). WHENEVER A NEW PROJECT IS STARTED, THE TOP PART SHOULD BE FILLED IN. A COPY SHOULD BE RETURNED TO THE DATA MANAGEMENT CENTRAL FILE, KEEP THE ORIGINAL FOR YOUR RECORDS TO KEEP TRACK OF WHAT HAS BEEN PROVIDED. IMSG WILL CHECK OFF WHEN ALL INFORMATION IS RECEIVED

FOLLOWING THIS PROCESS WILL IMPROVE THE FOLLOWING:

- TURN-AROUND TIME FOR DELIVERABLES NEEDED WHEN ALL RESULTS HAVE BEEN RECEIVED.
- CONFIDENCE THAT ALL SAMPLE RESULTS HAVE BEEN RECEIVED
- CONSISTENCY OF SAMPLE NOMENCLATURE
- CORRECTNESS OF SAMPLE ATTRIBUTES
- REVIEW OF INVOICES
- ENABLE IMSG PERSONNEL TO BETTER TRACK UPCOMING WORKLOAD

PROJECT START-UP CHECKLIST

INFORMATION NEEDED TO CREATE NEW DATABASE

PROJECT NAME:__NSWC Crane___

CTO #:_166__ JOB #:__N3994____ ETS Code:__ Not applicable_____

PROJECT MANAGER/CONTACT:_____R. Basinski_____

LABELS: Y / N DUE DATE:__TBD___

VALIDATE: Y / N / L DUE DATE:__TBD___

COMBINE WITH HISTORICAL DATA: Y/N

SAMPLE DATA CHECKLIST:

__x__ SAMPLE NUMBERS AND ANALYSES (LOCATIONS,DEPTHS)

__x__ SECTION OF WORK PLAN PERTAINING TO SAMPLE NOMENCLATURE

__x__ LABORATORY/BOTTLE REQUIREMENTS

__x__ LAB SPECS

__x__ COC'S

__x__ SAMPLE LOG SHEETS

__x__ DUPLICATE ID'S / ORIGINALS

__x__ SURVEY DATA / SAMPLE LOCATION MAPS

__x__ BREAKDOWN OF PROJECT BY SITE / MATRIX FOR FUTURE PRINTOUTS

__x__ TABLE HEADERS (SEE EXAMPLE)

TO BE COMPLETED BY IMSG:

_____ FINAL RESULTS GIVEN TO _____ (PM/IMSG)

DATE:_____

_____ SAMPLE DATA LOADED INTO NEW/EXISTING PROJECT DATABASE

_____ RESULTS LOADED INTO NEW/EXISTING PROJECT DATABASE

PATHNAME OF PROJECT DATABASE:_____

_____ DATA LOADED INTO GIS

ATTACHMENT F5

SAMPLE TRACKING DATABASE EXAMPLE

CTO 020 SDG U06972

Sdg	Sample Number	Lab id	Fraction	Sort	Lab Rec	Rec Date	Turn-time	Laboratory
F5717	H10MW0501	F5717-2	M	M	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	MISC	CL	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	MISC	NTA	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	MISC	NTI	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	MISC	SO4	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	MISC	SUL	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	OS	OS	03-Feb-00	28-Feb-00	25	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	OV	ETHA	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	OV	ETHE	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	OV	METH	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	OV	OV	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ
F5717	H10MW0501	F5717-2	PAH	PAH	25-Jan-00	28-Feb-00	34	ACCUTEST, NJ

ATTACHMENT F6

EXAMPLE SAMPLE JAR LABELS

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: CTO 038 Location: NSWC CRANE	
Sample No: BGSBP0401		Tag # : A0001	
Date: (1)	Time: (2)	Preserve:	
Analysis: TAL Metals + Tin		Matrix: SOIL	
Sampled By: (3)		Laboratory: LAUCKS	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Location:	
Sample No:		Tag # :	
Date: (1)	Time: (2)	Preserve:	
Analysis:		Matrix:	
Sampled By:		Laboratory:	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: CTO 038 Location: NSWC CRANE	
Sample No: BGSBP0401		Tag # : A0001	
Date: (1)	Time: (2)	Preserve:	
Analysis: TAL Metals + Tin		Matrix: SOIL	
Sampled By: (3)		Laboratory: LAUCKS	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Location:	
Sample No:		Tag # :	
Date: (1)	Time: (2)	Preserve:	
Analysis:		Matrix:	
Sampled By:		Laboratory:	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: CTO 038 Location: NSWC CRANE	
Sample No: BGSBP0401		Tag # : A0001	
Date: (1)	Time: (2)	Preserve:	
Analysis: TAL Metals + Tin		Matrix: SOIL	
Sampled By: (3)		Laboratory: LAUCKS	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Location:	
Sample No:		Tag # :	
Date: (1)	Time: (2)	Preserve:	
Analysis:		Matrix:	
Sampled By:		Laboratory:	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Location:	
Sample No:		Tag # :	
Date: (1)	Time: (2)	Preserve:	
Analysis:		Matrix:	
Sampled By: (3)		Laboratory:	

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project: Location:	
Sample No:		Tag # :	
Date: (1)	Time: (2)	Preserve:	
Analysis:		Matrix:	
Sampled By: (3)		Laboratory:	

ATTACHMENT F7

ELECTRONIC DATA DELIVERABLE REQUIREMENTS FOR ANALYTICAL LABORATORIES

ELECTRONIC DATA FORMAT REQUIREMENTS

1.0 INTRODUCTION

The laboratory is to provide 3.5" high-density diskette(s) containing separate database (DBF) files in the format specified in this attachment. The electronic deliverable includes all environmental samples, sample dilutions, sample reanalyses, and laboratory quality control samples. **All entries in the electronic deliverable must agree exactly with the final entries reported on the hard-copy data package sample result summaries**. Any corrections made to the hard-copy data must also be made to the electronic file. Appropriate qualifiers as identified by the analytical protocol must also be designated; laboratory QC non-compliance codes are not to be depicted.

Each diskette is to be properly labeled with the laboratory name, project name, file name(s), and laboratory point of contact. Electronic files should be delivered in the same fashion as the hard-copy data packages. A separate .dbf file will be made for each analytical fraction (by method) and each sample delivery group (SDG). The files will be named so the first character is the analytical fraction designator, followed by an underscore, followed by the SDG name. For example, the file for the volatile fraction for SDG BR001 should be named V_BR001.DBF. Additionally, the laboratory must provide a hard-copy list of all electronic files saved to the diskette, indicating what analytical fraction and matrix the file data contained therein pertain to. All electronic data deliverables are due within the same time established for the associated hard-copy data packages.

In addition, the laboratory QC officer must read and sign a copy of the Quality Assurance Review Form displayed on the next page of this attachment. Electronic deliverables are not considered to be complete without the accompanying Quality Assurance Review Form.

I _____, as the designated Quality Assurance Officer, hereby attest that all electronic deliverables have been thoroughly reviewed and are in agreement with the associated hard-copy data. The enclosed electronic files have been reviewed for accuracy (including significant figures), completeness, and format. The laboratory will be responsible for any labor time necessary to correct enclosed electronic deliverables that have been found to be in error. I can be reached at (____)_____if there are any questions or problems with the enclosed electronic deliverables.

Signature:_____ Title: _____ Date: _____

The analytical data will be delivered electronically in a Dbase III file format (filename.dbf). The exact structure of the database is described in the table below. It will be the responsibility of the laboratory to ensure that all electronic entries are in strict accordance with the information provided on the Form I.

An example database will be sent for review before to the first electronic deliverable in Dbase III format. The example file will be examined for completeness and comments will be sent to the laboratory. Any questions regarding the electronic deliverable will be directed to Andy Kendrick at Tetra Tech NUS, (412) 921-8623.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
SAMPLE_NO	C	25	Field sample ID as listed on the chain-of-custody. The sample number indicated in this field should never be truncated. The only exception for this field not matching the chain-of-custody is for reanalyses and matrix spike results in which a RE or MS suffix will be added to the sample number respectively.
TRUNCATE	C	15	If the field sample ID listed on the chain-of-custody is truncated by the laboratory for use with the laboratory software, the truncated sample ID should appear in this field.
LAB_ID	C	15	Laboratory number for the given sample.
LABORATORY	C	25	Laboratory name.
BATCH_NO	C	10	Laboratory code for batch of samples included in a given run.
ASSOC_BLNK	C	15	Laboratory name of the method blank associated with that particular batch of samples.
QC_TYPE	C	15	Normal Environmental Sample = NORMAL, Laboratory Duplicate = DUPLICATE, Matrix Spike = MS, Matrix Spike Duplicate = MSD, Laboratory Control Sample = LCS, Laboratory Control Sample Duplicate = LCSD, Method Blank = M_BLANK, Preparation Blank = P_BLANK.
SAMP_DATE	D	8	Date of sample collection as indicated on the chain-of-custody. Example: 11/07/93.
REC_DATE	D	8	Date sample was received by the laboratory.
EXTR_DATE	D	8	Date sample was extracted or prepared by the laboratory.
ANAL_DATE	D	8	Date sample was analyzed by the laboratory.
RUN_NUMBER	N	2 (0)	The number of the analytical run for a given sample in sequence. For example, if a sample is diluted and reanalyzed, the original run number would be 1 and the reanalysis would be 2.
SDG	C	15	Sample delivery group identifier assigned by the laboratory. This number should exactly match the SDG designated on the hard-copy data package.

DATA FIELD	DATA TYPE	FIELD WIDTH	DATA FIELD DESCRIPTION
PROJECT_NO	C	10	Identification of project number or CLEAN Task Order (CTO) number.
PROJ_MNGR	C	25	The Tetra Tech NUS project manager's last name, followed by a comma, followed by the first initial of the project manager (e.g. Hutson, D).
PARAMETER	C	45	Chemical or analyte name exactly as reported on Form I.
CAS_NO	C	10	Chemical Abstract Service number for the parameter listed. The CAS number should be reported exactly as it is listed in publications such as the Merck Index. This field should be left blank for those parameters not having CAS numbers (e.g., Total Organic Carbon).
FRACTION	C	5	Metals = M, Volatiles = OV, Semivolatiles/BNAs = OS, Pesticides = PEST, Herbicides = HERB, Polychlorinated Biphenyls = PCB, Explosives = EXP, Any petroleum hydrocarbon or fuel = TPH, Wet Chemistry = WET, Radionuclide = RAD, Miscellaneous = MISC
METHOD	C	20	Analytical method used to quantitate parameter concentrations as listed in the laboratory technical specification (e.g., 8270A for SW-846 Method 8270A.)
LAB_RESULT	N	20 (6)	Reported value in units specified in the UNITS field containing the proper number of significant digits. The % Recovery will be placed in this field for matrix spike and laboratory control sample results.
UNITS	C	5	The units of measure as reported on the Form I.
LAB_QUAL	C	2	The laboratory qualifier as reported on the Form I. For example, a U qualifier should be used for all nondetected results.
IDL	N	15 (6)	Instrument detection limit in units specified in the UNITS field.
MDL	N	15 (6)	Method detection limit in units specified in the UNITS field and method specified in the METHOD field.
CRDL_CRQL	N	15 (6)	Contract Required Detection/Quantitation Limit in the units specified in the UNITS field. RDL for non-CLP parameters.
DIL_FACTOR	N	6 (1)	Dilution factor.
PCT_MOIST	N	5 (1)	Percent moisture for soil samples; blank for water samples.
COMMENTS	C	20	Analytical result qualifier or comment other than that listed in the LAB_QUAL field. Example: Reanalysis.

C = Character string (everything shall be reported in capital letters).
N = Numeric string (decimal places are in parentheses in field width column).
D = Date (Ex: 05/25/97).

ATTACHMENT F8

DATABASE STRUCTURE

DATA DICTIONARY

TETRA TECH NUS DATABASE STANDARDS

January, 1999

DRAFT

TABLE: well

PRIMARY KEY: location

Table Structure

FIELD	DESCRIPTION
location	Unique location name.
post_id	Location name as derived from original source document.
instal_date	Date the monitoring well was installed. Null for other location types.
loc_type	Type of location (e.g., soil boring, GW well, drive point, wipe)
Northing	Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
Easting	Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
horiz_datum	Datum in which the horizontal coordinates were derived.
grnd_surf	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field.
vert_datum	Datum in which the vertical coordinates were derived.
datum_state	State for which datum was developed.
surveyed	Logical field denoting whether positional data were surveyed or digitized.
Surveyor	Company who performed the survey.
survey_date	Date in which survey was performed.
surv_method	Surveying method used.
longitude	Longitude.
latitude	Latitude.

Table Indexes

INDEX	TYPE
location	Primary
hd	Regular
vd	Regular
loc_type	Regular

Table Relations:

Relation 1
*RelatedChild loc_type
*RelatedTable loc_type_vvl
*RelatedTag loc_type

Relation 2
*RelatedChild cd
*RelatedTable coord_datum_vvl
*RelatedTag cd

TABLE: loc_type_vvl - Valid value list for LOC_TYPE field in the well table.
PRIMARY KEY: loc_type

Table Structure

FIELD	DESCRIPTION
loc_type	Location type
description	Description of location type

Table Indexes

INDEX	TYPE
loc_type	Primary

TABLE: coord_datum_vvl - Valid value list for HORIZ_DATUM field in the well table.
PRIMARY KEY: cd (coord_datum)

Table Structure

FIELD	DESCRIPTION
coord_datum	Datum in which coordinates reflect.

Table Indexes

INDEX	TYPE
cd (coord_datum)	Primary

TABLE: sample_data - Sample data table
PRIMARY KEY: nsample

Table Structure

FIELD	DESCRIPTION
location	Unique location name.
Matrix	Sample matrix.
nsample	Unique sample identification.
sample	Sample identification as designated on Chain-of-Custody.
sacode	Sample code for reference to field duplicates.
top_depth	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
Bottom_depth	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. *Rule Expression*: if(bottom_depth>0,top_depth<=bottom_depth)
qc_type	Quality control type.
status	Status of sample location - Normal or excavated.

sample_date	Date in which sample was collected.
validated	Logical field denoting whether or not data validation was performed on sample.
coll_method	Sample collection method.
cto_proj	Clean task order (Navy) or project number in which the sample was collected (e.g., 129).
proj_manager	Internal project manager for which the data were originally generated (e.g. Hutson, D.).

Table Indexes

INDEX	TYPE
location	Regular
nsample	Primary
sacode	Regular
matrix	Regular
status	Regular
qc_type	Regular
coll_meth	Regular

Table Relations:

Relation 1
*RelatedChild sacode
*RelatedTable sacode_vvl
*RelatedTag sacode
Relation 2
*RelatedChild qc_type
*RelatedTable qc_type_vvl
*RelatedTag qc_type
Relation 3
*RelatedChild matrix
*RelatedTable matrix_vvl
*RelatedTag matrix
Relation 4
*RelatedChild location
*RelatedTable well
*RelatedTag location
Relation 5
*RelatedChild coll_meth
*RelatedTable coll_method_vvl
*RelatedTag coll_meth

TABLE: sacode_vvl - Sample code valid value list for SACODE field in sample_data.dbf
PRIMARY KEY: sacode

Table Structure

FIELD	DESCRIPTION
sacode	Sample code designating whether sample is a normal environmental sample, a field duplicate, or the average of field duplicate pairs.
description	Description of sacode entry.

Table Indexes

INDEX	TYPE
sacode	Primary

TABLE: qc_type_vvl - Quality control valid value list for QC_TYPE field in sample_data.dbf
PRIMARY KEY: qc_type

Table Structure

FIELD	DESCRIPTION
qc_type	Quality control type.
description	Description of quality control type.

Table Indexes

INDEX	TYPE
qc_type	Primary

TABLE: matrix_vvl - Matrix valid value list for MATRIX field in sample_data.dbf
PRIMARY KEY: matrix

Table Structure

FIELD	DESCRIPTION
matrix	Sample matrix.
description	Description of sample matrix code.

Table Indexes

INDEX	TYPE
matrix	Primary

TABLE: well_completion
PRIMARY KEY: None

Table Structure

FIELD	DESCRIPTION
location	Unique location name.
top_casing	Elevation of top of well casing in vertical datum found in VERT_DATUM in the well table.
hole_diameter	Diameter of the drilled hole in inches. *Rule Expression*: hole_diameter>casing_id.AND.hole_diameter>casing_od
scr_aquifer	Aquifer name in which the screen resides.
screen_material	Type of material from which the screen is constructed.
scrn_slot_size	Screen slot size in thousandths of an inch.
scrn_top_depth	Depth below ground surface to the top of the screen (in feet).
scrn_bot_depth	Depth below ground surface to the bottom of the screen. *Rule Expression*: if(scrn_bot_depth>0,scrn_top_depth<scrn_bot_depth)
scrn_top_elev	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table.
scrn_bot_elev	Elevation the top of the screen in vertical datum found in VERT_DATUM in the well table. *Rule Expression*: if(scrn_bot_elev>0,scrn_top_depth>scrn_bot_depth)
drill_method	Drilling method for well installation.
contractor	Drilling contractor.
casing_material	Type of material from which the casing is constructed.
depth_to_seal	Depth below ground surface to seal (in feet).
seal_material	Type of material from which the seal is constructed.
fill_top_depth	Depth below ground surface to the top of fill material (in feet).
fill_bot_depth	Depth below ground surface to the bottom of fill material (in feet). *Rule Expression*: if(fill_bot_depth>0,fill_top_depth<scrn_bot_depth)
fill_material	Type of material used for fill.
comments	Geologist's comments.

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location
TABLE: lithology
PRIMARY KEY: None

FIELD	DESCRIPTION
location	Unique location name.
top_lithology	Depth in feet below ground surface to the top of lithologic unit.
bottom_lithology	Depth in feet below ground surface to the bottom of lithologic unit.
uscs_code	Unified Soil Classification System for lithology type.
blow_counts	Number of blow counts recorded on boring log.
description	Geologist's description of lithology.
comments	Geologist's comments.

Table Indexes

INDEX	TYPE
location	Regular
uscs_code	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location
Relation 2
*RelatedChild uscs_code
*RelatedTable lithology_vvl
*RelatedTag uscs_code

TABLE: lithology_vvl - Lithology valid value list for USCS_CODE field in lithology.dbf
PRIMARY KEY: uscs_code

Table Structure

FIELD	DESCRIPTION
uscs_code	Unified Soil Classification System for lithology type.
descript	Description of lithology for given USCS code.

Table Indexes

INDEX	TYPE
uscs_code	Primary

TABLE: coll_method_vvl - Collection method valid value list for COLL_METHOD field in sample_data.dbf
PRIMARY KEY: coll_meth

Table Structure

FIELD	DESCRIPTION
coll_method	Sample collection method.

Table Indexes

INDEX	TYPE
coll_method	Primary

TABLE: cas_vvl - CAS number valid value list for CAS field in analytical results.dbf
PRIMARY KEY: cas

Table Structure

FIELD	DESCRIPTION
parameter	Parameter or chemical name.
cas	Chemical Abstracts Service Number.

Table Indexes

INDEX	TYPE
parameter	Regular
cas	Primary

TABLE: analytic_results
PRIMARY KEY: nfp (nsample+fraction+parameter)

Table Structure

FIELD	DESCRIPTION
nsample	Unique sample identification.
lab_id	Laboratory sample identification.
laboratory	Laboratory name.
batch_no	Analytical batch number.
assoc_blnk	Associated blank.
extr_date	Extraction date.
anal_date	Analysis date.
run_number	Sequential analytical run number.
sdg	Sample delivery group.
parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate).

cas	Chemical Abstracts Service Number.
fraction	Analtytical fraction.
method	Analytical method.
lab_result	Analytical result as reported by the laboratory.
lab_qual	Qualifier as reported by the laboratory.
val_res	Final result (via validation or otherwise).
result	Final analytical result with the correct number of significant figures.
val_qual	Validation qualifer (null if data were not validated).
qual	Final qualifer (validation or otherwise).
qual_code	Validation flag used to define the quality control noncompliance.
units	Units of measure for the RESULT field.
idl	Instrument detection limit (same units as UNITS field).
mdl	Method detection limit (same units as UNITS field).
crdl_crql	Contract required detection/quantitation limit (same units as UNITS field).
dil_factor	Dilution factor.
pct_moist	Percent moisture.
comments	Comments from laboratory analyst.

Table Indexes

INDEX	TYPE
nfp	Primary
units	Regular
qual	Regular
fraction	Regular
parameter	Regular
nsample	Regular
cas	Regular

Table Relations:
Relation 1
*RelatedChild cas
*RelatedTable cas_vvl
*RelatedTag cas
Relation 2
*RelatedChild units
*RelatedTable units_vvl
*RelatedTag units
Relation 3
*RelatedChild qual
*RelatedTable qual_vvl
*RelatedTag qual
Relation 4

*RelatedChild fraction
*RelatedTable fraction_vvl
*RelatedTag fraction
Relation 5
*RelatedChild parameter
*RelatedTable para_vvl
*RelatedTag para
Relation 6
*RelatedChild nsample
*RelatedTable sample_data
*RelatedTag nsample

TABLE: units_vvl - Units valid value list for UNITS field in analytical_results.dbf
PRIMARY KEY: Units

Table Structure

FIELD	DESCRIPTION
units	Units of measure for chemical analysis.
description	Description of units.

Table Indexes

INDEX	TYPE
units	Primary

TABLE: qual_vvl
PRIMARY KEY: qual - Qualifier valid value list for QUAL field in analtyic_results.dbf

Table Structure

FIELD	DESCRIPTION
qual	Final QA qualifier.
description	Definition of qualifier.

Table Indexes

INDEX	TYPE
qual	Primary

TABLE: fraction_vvl - Analytical fraction valid value list for FRACTION field in analytic_results.dbf
PRIMARY KEY: fraction

Table Structure

FIELD	DESCRIPTION
fraction	Analytical fraction.
description	Description of fraction.

Table Indexes

INDEX	TYPE
fraction	Primary

TABLE: para_vvl
PRIMARY KEY: parameter

Table Structure

FIELD	DESCRIPTION
para	Parameter or chemical name.
frac_name	Analytical fraction for given parameter.

Table Indexes

INDEX	TYPE
para	Primary

TABLE: fluid
PRIMARY KEY: None

Table Structure

FIELD	DESCRIPTION
location	Unique location name.
meas_elev	Measuring point elevation.
dep_to_water	Depth below ground surface to water table (in feet).
dep_to_fp	Depth below ground surface to free product (in feet).
elev_water	Elevation of water level.
elev_fp	Elevation of free product.
prod_thick	Product thickness in feet.
meas_date	Date measurement was taken.

Table Indexes

INDEX	TYPE
location	Regular

Table Relations:
Relation 1
*RelatedChild location
*RelatedTable well
*RelatedTag location

ATTACHMENT F9

ARCVIEW GIS STRUCTURE

ARCVIEW GIS STRUCTURE

The project ArcView GIS shall have the following directory structure and database table structure.

PART ONE: DIRECTORY STRUCTURE

The following table defines the directory structure and major file names/types located within each directory.

Main subdirectory	First tier subdirectories	Second tier subdirectories	Files/Types
p:\gis\project name\	database\		coordinate.dbf cross_reference.dbf res_ gw.dbf res_so.dbf res_sd.dbf res_sw.dbf well_completion.dbf
		criteria\	crit_ gw.dbf crit_so.dbf crit_ sd.dbf crit_sw.dbf crit_des.dbf
	mapping\	aerial\	registered aerial photos
		drg\	USGS Digital Raster Graphic
		image\	GeoStatistic Layers, pictures of sites, equipment, EVS, and all other raster files.
		dwg\	AutoCAD files
		dgn\	Microstation files
		shp\	samp_gw.shp .dbf .shx samp_so.shp .dbf .shx samp_sd.shp .dbf .shx samp_sw.shp .dbf .shx and all other AV shape files
	working\	database\	Files used to generate specific drawings will be put under the working subdirectory in subdirectories similar to database and mapping. These will not be included in CD deliverable.
		mapping\	same as above

PART TWO: DATABASE TABLE STRUCTURE

The project ArcView GIS will contain separate database tables to store analytical, criteria, and coordinate information. The structure of these tables is presented below.

Analytical Data Table

The following table lists all the fields contained in the analytic database table.

FIELD	VISIBLE	ALIAS*	DESCRIPTION
site	Yes	Site or SWMU	Site or SWMU.
location	Yes	Location	Unique location name.
nsample	Yes	Sample	Unique sample identification.
sample	No		Sample identification as designated on chain-of-custody.
sample_date	Yes	Sample Date	Date on which sample was collected.
matrix	Yes	Matrix	Sample matrix.
sacode	Yes	Sample Code	Sample code for reference to field duplicates.
depth	Yes	Depth	Depth in feet to the middle of the sample interval. Applicable for soil and sediment samples.
top_depth	Yes	Top Depth	Depth in feet to the top of the sample interval. Applicable for soil and sediment samples.
bottom_depth	Yes	Bottom Depth	Depth in feet to the bottom of the sample interval. Applicable for soil and sediment samples. Rule Expression: if(bottom_depth>0,top_depth<=bottom_depth)
parameter	Yes	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate).
cas	Yes	CAS	Chemical Abstracts Service Number.
fraction	Yes	Fraction	Analytical fraction.
val_res	Yes	Numeric Result	Final result (via validation or otherwise).
qual	Yes	Qualifier	Final qualifier (validation or otherwise).
units	Yes	Units	Units of measure for the RESULT field.
method	Yes	Method	Analytical method.
status	Yes	Status	Status of sample location – Normal or excavated.
validated	Yes	Validated	Logical field denoting whether or not data validation was performed on sample.

FIELD	VISIBLE	ALIAS*	DESCRIPTION
coll_method	Yes	Collection Method	Sample collection method (e.g., grab/composite).
cto_proj	Yes	CTO	Clean task order (Navy) or project number for which the sample was collected (e.g. 129).
proj_manager	Yes	Project Manager	Internal project manager for which the data were originally generated (e.g. Hooper, P.).
lab_id	No	Laboratory ID	Laboratory sample identification.
laboratory	No	Laboratory	Laboratory name.
batch_no	No	Batch Number	Analytical batch number.
assoc_blnk	No	Associated Blank	Associated blank.
extr_date	No	Extraction Date	Extraction date.
anal_date	No	Analysis date	Analysis date.
run_number	No	Run Number	Sequential analytical run number.
sdg	No	SDG	Sample delivery group.
lab_result	No	Result	Analytical result as reported by the laboratory.
lab_qual	No	Lab Qualifier	Qualifier as reported by the laboratory.
result	No	String Result	Final analytical result with the correct number of significant figures.
val_qual	No	Validation Qualifier	Validation qualifier (null if data were not validated).
idl	No	Detection Limit	Instrument detection limit (same units as UNITS field).
mdl	No	Detection Units	Method detection limit (same units as UNITS field).
crdl_crql	No		Contract required detection/quantitation limit (same units as UNITS field).
dil_factor	No	Dilution factor	Dilution factor.
pct_moist	No	Percent moisture	Percent moisture.
ourresult	No		
qc_type	No		Quality control type.
comments	No	Comments	Comments from laboratory analyst.

*A blank indicates that no alias exists.

Criteria Table

Each medium will have a criteria table to specify the applicable criteria for all parameters.

FIELD	ALIAS	DESCRIPTION
parameter	Parameter	Parameter or chemical name (using IUPAC nomenclature where appropriate).
epa_mcl	None	Federal MCL – groundwater.

Note: Usually there will be many criteria fields. This example table only shows the "epa_mcl" criteria field.

Criteria Description Table

This table stores the definition or description of all standards and criteria used in the project. For example, epa_mcl's media would be GW, description would be "Federal Maximum Contaminant Level."

FIELD	Visible	DESCRIPTION
Field	Yes	
Media	Yes	
Descript	Yes	

Coordinate Table

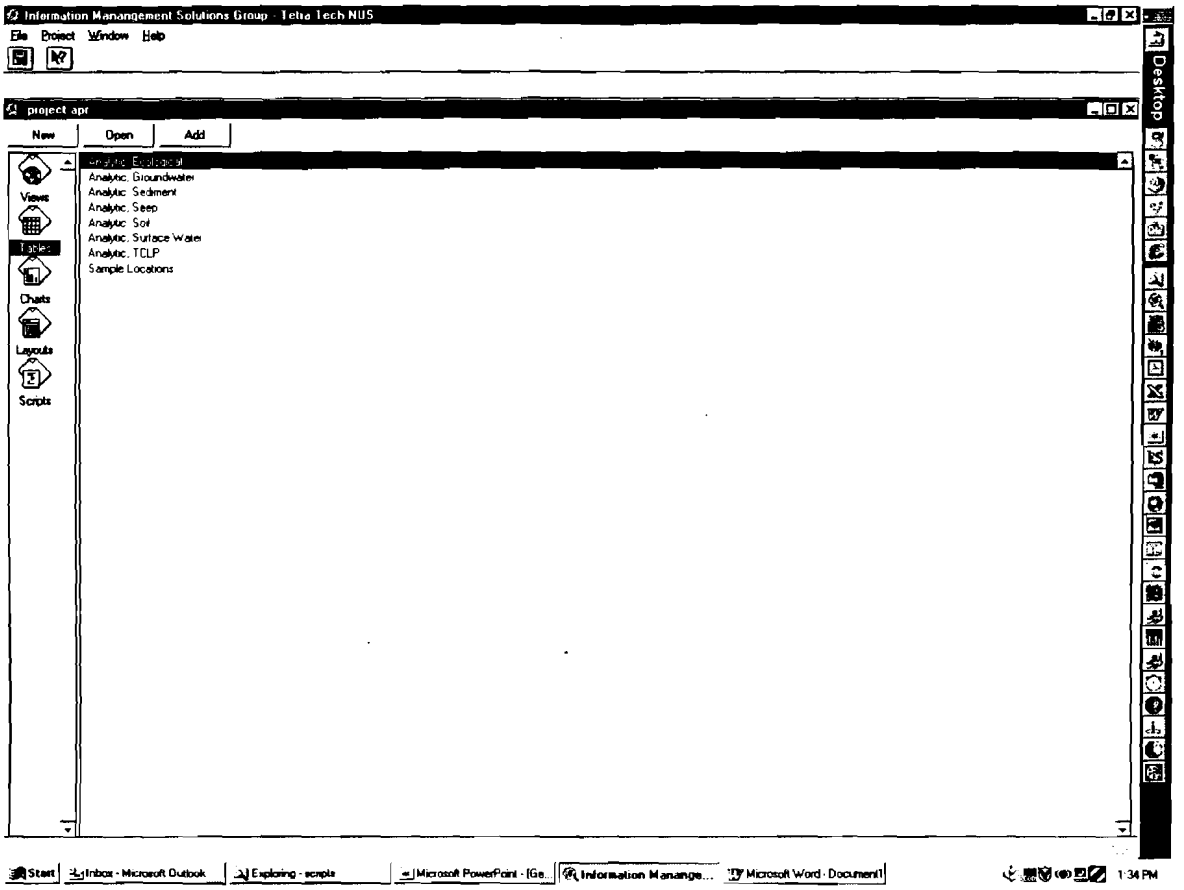
The coordinate table holds all the geographic position information of sampling locations

FIELD	Visible	ALIAS*	DESCRIPTION
location	Yes		Unique location name.
post_id	Yes	Location Designation	Location name as derived from original source document.
instal_date	No	Installation Date	Date the monitoring well was installed. Null for other location types.
loc_type	Yes	Location Type	Type of location. Example MW, HP, etc.
northing	Yes		Northing coordinate in horizontal datum referenced in the HORIZ_DATUM field.
easting	Yes		Easting coordinate in horizontal datum referenced in the HORIZ_DATUM field.
grnd_surf	Yes	Ground Surface Elevation	Ground surface elevation with reference to mean sea level in vertical datum referenced in the VERT_DATUM field
horiz_datum	Yes	Horizontal Datum	Datum in which the horizontal coordinates were derived.
vert_datum	Yes	Vertical Datum	Datum in which the vertical coordinates were derived.

FIELD	Visible	ALIAS*	DESCRIPTION
fatum_state	Yes	Coordinate System	State for which datum was developed.
durveyed	Yes		Logical field denoting whether positional data were surveyed or digitized.
durveyor	Yes		Company who performed the survey.
durvey_date	No	Survey Date	Date on which survey was performed.
durv_method	No	Survey Method	Surveying method used.
longitude	No		Longitude.
latitude	No		Latitude.
gw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
sd_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
so_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
sw_code	Yes		This will be populated by database personnel. It will be used for event driven theme.
_nullflags	No		Various fields are put in by database personnel starting here and followed by several fields. Make all of these invisible.

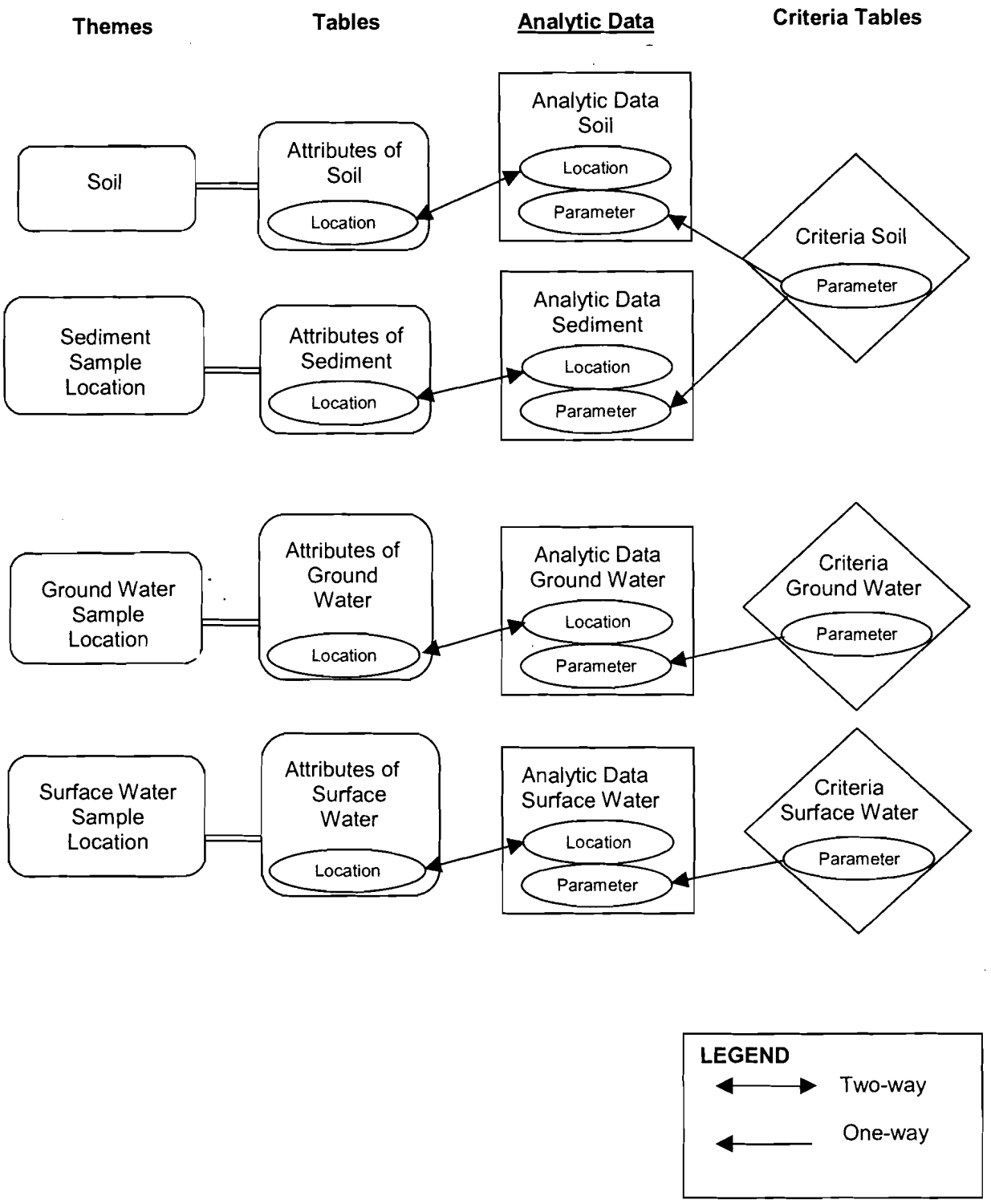
*A blank indicates that no alias exists.

TABLE LINKS



All sample location theme attribute tables are two-way linked to the corresponding analytical data table. In addition, the analytical data tables are joined to the criteria table. The following diagram illustrates the relationship.

Database Model:



APPENDIX G

FIELD AUDIT CHECKLIST

Location: NSWC Crane, Crane, Indiana

Project: NSWC Crane Phase III RFI Of SWMUS 12, 13, 16, and 19, CTO 166

Date of Audit: _____

Instructions: Record answers to questions below, providing comments as required for clarification.

QA/QC Procedures

1. Were any field observations, deficiencies, non-conformances, or complaints recorded by the site QA/QC Officer or other personnel?
   If so, summarize below.

   _____
   _____
   _____
   _____
   _____

2. Based on personnel interviews, did any variances from the project planning documents occur? If so, what were they?

   _____
   _____
   _____
   _____

3. Were field modification records pertinent to the above initiated in an appropriate manner?

   _____

4. If applicable, were corrective action plans implemented (according to proper procedure) regarding Question 2?

   _____
   _____
   _____

5. Were field QC samples obtained with the frequency specified in the QAPP?

   _____

6. For this site, were field duplicates submitted "blind" to the laboratory?

7. For this site, are sufficient replicate aliquots of samples designated for the laboratory for the matrix spike/duplicate analyses specified in the QAPP?

Boring/Drilling

8. Is the drilling method specified in the QAPP being used?

9. In accordance with TtNUS policies and field SOPs, the FOL has the authority to change drilling methods if site conditions so dictate. Did any change in drilling methods from that cited in the project planning documents occur? If so, discuss.

10. If a change in drilling methods was required, did the FOL properly document the change and the rationale for the change?

11. Were any field changes initiated by the drilling subcontractor? If so, was the FOL notified and were the changes documented?

12. Was the air rotary rig outfitted with a filter on the compressed line?

_____
_____
_____
_____

13. Have all abandoned borings been backfilled as specified in the QAPP or applicable SOP?

_____
_____

14. When applicable, was the casing cleaned before sampling? (In most cases, an inch or two of cuttings may be left in the borehole with little or no problem. However, if more than a few inches for cuttings are encountered, the borehole must be recleaned prior to attempting sampling.)

water wash (disturbed samples above and below water table) _____
clean-out auger (undisturbed samples below water table) _____
dry method (undisturbed samples above water table) _____

15. Were any drilling lubricants used? If so, were the procedures cited in TtNUS SOP CTO 166-12 observed?

_____
_____
_____

16. Were detailed boring logs maintained by the site geologist for each borehole? (Logging is not required if explicitly stated so in the associated FSAP.)

_____
_____
_____
_____

17. Was the following information complete on the borehole logs:
description of materials _____
description of samples _____
sampling method _____
blow counts _____
final location for drilling _____

Soil Sampling

18. Are the sampling devices and boring methods designated in the QAPP or applicable project-specific SOP being used?

_____
_____
_____
_____

19. Was the following information recorded in the boring logs or the field notebook?

For soil classification:

Was the USCS classification and soil type (clay, silt, sand) indicated?

_____

Were the following characteristics identified per the relevant TtNUS SOP CTO166-07 sections?

color _____
soil type _____
relative density and consistency _____
stratification _____
texture/fabric/bedding _____

20. For surface soil samples obtained by hand auger or scoop or trowel, were the following activities performed?

area cleared of loose debris prior to sampling _____
location marked with numbered stake or pin flag _____
sketch of approximate locations of sample points in site notebook _____

21. Were soil samples NOT collected from the top 6" soil interval? Yes/No

Groundwater Sampling

22. Were all monitoring wells properly developed, purged and recovered prior to sampling?

_____
_____

23. When applicable, were well volumes calculated as described in SOP CTO166-16?

_____

24. If a peristaltic pump was used to obtain Volatile Organic Compound (VOC) samples, was it verified that no degassing "bubbles" developed?

_____

25. If samples were acquired by a pump, was the pump lowered to midscreen (middle of open section of uncased wells) for sample acquisition?

_____

26. If samples were collected using bailers, were only bailers equipped with check balls used?

_____

27. For samples acquired by packer assembly, was the packer positioned just above the screen (or open section for uncased wells), prior to inflating?

_____

Surface Water and Sediment Sampling

28. In accordance with SOP 166-19, surface water samples will be taken downstream and will proceed upstream, was the above rule observed?

_____
_____

29. Per SOP CTO166-19; it is preferable to sample with the mouth facing upstream, was this practice observed?

_____
_____

30. Were the appropriate field parameters collected and recorded in accordance with SOP CTO 166-19?

_____
_____
_____
_____

Equipment Decontamination Procedures

31. Has an adequate, pre-determined area for steam cleaning of equipment been established?

_____

_____

32. Is the decontamination (decon) area lined and/or bermed?

_____

_____

33. Is sampling equipment decontaminated as required?

_____

_____

34. Was steam cleaning conducted:

prior to commencement of field activities? _____
between boring/pit locations? _____
at the end of field activities?_____

35. Verify that all sampling equipment not subject to steam cleaning (e.g., trowels, mixing bowls, etc.) are subjected to decontamination per the sequence outlined in the QAPP and project-specific SOP

_____

_____

Calibration and Use of Field Monitoring Equipment

36. Were the following calibration criteria observed:

calibration according to manufacturer's instructions _____
calibration only by qualified individuals _____
calibrated and operationally checked prior to project assignment _____
use of certified/traceable standards _____
calibration documented _____
if applicable, maintenance documented _____

37. For Photoionization Detectors (PIDs), is the proper eV Lamp (e.g., 9.5, 10.2, 11.7) installed?

38. Confirm that proper PID Start-up and Shut-down procedures are performed as required.

39. Has PID UV light source window cleaning been conducted as required?

40. Has the PID ionization chamber been cleaned as required?

41. Has the PID unit been recharged after every use?

Waste Handling Procedures

42. Were cuttings or fluids disposed of in accordance with the QAPP Section 4 requirements (i.e., discharged to ground, drummed, or tanked)?

43. Do the project planning documents provide for the disposal of Personal Protective Equipment (PPE) by double-bagging and discard?

44. By what method are PPE disposed of?

45. If applicable, were used spill-containment materials containerized or otherwise acceptably disposed of?

_____

_____

Sample Handling

46. Are the appropriate containers provided by the laboratory being used for each sample?

_____

_____

_____

47. Has the temperature blank been handled properly and one submitted with each cooler of samples?

_____

_____

48. Have equipment rinsate blanks of the proper type and frequency been obtained?

_____

_____

49. Have source water blanks been obtained from water sources applicable to the field effort?

_____

_____

50. Have the rinsate and source water blanks been designated for the same analyses as the associated samples?

_____

_____

51. Has sample custody been maintained with regard to the following criteria:

    A sample is under an individual's custody if:

    - it is in the individual's actual possession
    - it is in the individual's view after possession
    - it was locked up to prevent tampering
    - it was placed in a designated and identified secure area

    (The sample remains in the individual's custody until it is entrusted to a laboratory courier or commercial express carrier.)

_____

_____

Documentation

52. Are all sample logs complete (i.e., containing all information stipulated in SOPs CTO166-6, -19, -21, and -22)?

_____

_____

53. Have chain-of-custody (COC) forms been filled out for all samples, including field quality control samples?

_____

_____

54. Have the COC forms been signed by the appropriate individual at each step that the samples are relinquished?

_____

_____

55. Have the COC forms been filled-out using black waterproof ink?

_____

_____

56. If the COC form or other field document was corrected, was a line drawn through the information and was the change dated and initialed? (Use of white-out or erasure is not permitted.)

_____

_____

57. Have the appropriate analyses (per the QAPP) been properly designated for each sample on the COC form?

58. Have all sample labels been filled out appropriately and completely?

59. Have sample tags been properly completed and attached securely to samples?

60. Have all sample labels been filled out using indelible ink?

61. Do the sample identifications agree between the sample log, field notebook, sample label and COC form?

62. When applicable, have the name of the photographer, date, time, site location, and site description been entered sequentially into the site logbook as documentational photographs of the sampling have been taken?

63. Has the following information (at a minimum) been recorded in the site logbook:

- arrival/departure of site visitors
- arrival/departure of equipment
- start/completion of sampling activities
- health and safety issues

64. Is the site logbook a hard paginated, controlled distribution record bound book?

65. As required by SOP CTO166-03, does the cover of the site logbook contain the following information?

project name _____
project number _____
book number _____
start date _____
end date _____

66. Do the site logbook entries summarize the contents of other site notebooks where applicable?

_____

67. Have all site logbook entries been made in indelible ink?

_____
_____

68. If a logbook entry was corrected, was a line drawn through the information and was the change dated and initialed? (Use of white-out or erasure is not permitted.)

_____
_____

69. Did the Field Operations Leader sign all logbook pages utilized that day at the end of each day?

_____
_____

Auditor Name:_____

Auditor Signature:_____

Date:_____

APPENDIX H

FIELD STANDARD OPERATING PROCEDURES

SOP TABLE OF CONTENTS

SOP #	TITLE
CTO166-01	Sample Labeling
CTO166-02	Sample Identification Nomenclature
CTO166-03	Sample Custody and Documentation of Field Activities
CTO166-04	Borehole Advancement and Soil Coring Using Direct Push Technology
CTO166-05	Monitoring Well Sampling
CTO166-06	Calibration and Use of Photoionization Detector
CTO166-07	Borehole and Soil Sample Logging
CTO166-08	Surface and Subsurface Soil Sampling
CTO166-09	Inspection of Existing Monitoring Wells
CTO166-10	Monitoring Well Development
CTO166-11	Drilling and Geologic Logging of Boreholes in Rock
CTO166-12	Monitoring Well Installation
CTO166-13	Packer Testing of Uncased Boreholes
CTO166-14	Calibration and Care of Water Quality Meter
CTO166-15	Management of Investigation Derived Waste
CTO166-16	Low-Flow Well Purging and Stabilization
CTO166-17	Decontamination of Field Sampling Equipment
CTO166-18	Measurement of Water Levels in Monitoring Wells
CTO166-19	Surface Water Sampling
CTO166-20	Sediment Sampling
CTO166-21	Estimating Flow in Small Streams
CTO166-22	Hydraulic Conductivity Testing

STANDARD OPERATING PROCEDURE
NUMBER CTO166-01

SAMPLE LABELING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling and tagging sample containers. Sample labels and tags are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label and a sample tag will be attached to each sample container. The label and tag for each container will contain identical information.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil

Disposable medical-grade gloves (e.g. latex, nitrile)

Sample logsheets

Required sample containers: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.

Preprinted sample labels and sample tags

Chain-of-custody records

Sealable polyethylene bags

Heavy-duty cooler

Ice

3.0 PROCEDURES

3.1 The following information will be printed on the labels and tags prior to field activities.

- Project number (CTO 166)
- Project location (NSWC Crane)
- Sample ID
- Preservative

- Analysis to be performed
- Matrix type
- Laboratory name

3.2 Preprinted sample labels and tags will be prepared before the team mobilizes to the field. Check to determine if

- One sample label and tag exists for each sample container that is to be collected for all media during the field activities.
- The information printed on each tag and label is correct.
- Extra blank labels and tags are brought to the site in case additional environmental samples or QA samples are collected that are not anticipated in the QAPP. Additional blank labels and tags should also be brought to the site in case a sample container is broken or some of the preprinted labels or tags are accidentally lost before they are attached to a container.

3.3 Once at the field site, sample containers should have labels affixed before sampling activities begin.

3.4 Select the labeled containers that are appropriate for a given sample and fill in the date, time, and sampler's initials just before sampling begins. Use a black waterproof marker or pen.

3.5 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.

3.6 Write the same date, time, and sampler's initials on the sample tag as written on the label.

3.7 Place the sample container in a ziplock plastic bag and place in a cooler containing ice.

3.8 Fill in appropriate information on the Sample Collection Log Form and the Chain-of-Custody Form.

Example sample labels and tags are attached at the end of this SOP.

4.0 ATTACHMENTS

1. Sample Label and Tag

ATTACHMENT 1

SAMPLE LABEL AND TAG

SAMPLE LABEL AND TAG

166

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	**Project:** CTO-~~205~~ **Location:** NSWC CRANE

Sample No:		Tag # : A0001
Date:	Time:	Preserve:
Analysis: ~~PCBs~~		Matrix: SOIL
Sampled By:		Laboratory: LAUCKS

STRING

STANDARD OPERATING PROCEDURE
NUMBER CTO166-02

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Surface Warfare Center (NSWC). The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of identification and direct link to site and year

The NSWC Crane Environmental Protection Department must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with indelible ink
Sample tags
Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Monitoring Samples

All monitoring samples collected at NSWC Crane will be properly labeled with a sample label affixed to the sample container and a tag tied around the neck of the sample container. Each sample will be assigned a unique sample tracking number. The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated solid waste management unit (SWMU) or

associated site, sample type, location, and, for aqueous samples, where applicable, whether a sample is filtered, and/or the sample round number. For soil or sediment samples, the final four tracking numbers will identify the depth at which the soil or sediment sample was collected.

The alpha-numeric coding to be used in the NSWC Crane sample system is explained in the diagram and the subsequent definitions:

NN	**AA**	**A or N** **2 to 5-Characters**	**NN and/or A** **Aqueous only**	**NNNN** **Soils and Sediment only**
SWMU or Site Number	Sample Type	Location	Round Identifier and/or Filtered	Depth Interval

Character Type:

A = Alpha
N = Numeric

SWMU or Site Number:

12 = Mine Fill A
13 = Mine Fill B
16 = Cast High Explosives Fill/B146 Incinerator
19 = Pyrotechnic Test Area

Sample Type:

GW - Groundwater Sample from a Permanent Monitoring Well
ID - Investigation-Derived Waste Sample
SB - Soil Boring Sample
SD - Sediment Sample
SP - Seep Sample or Spring Sample
SS - Surface Soil Sample
SW - Surface Water Sample

Location:

The sample location code is the well number, soil sample location, sediment sample location, or the stream sample location (i.e., surface water, springs, or seeps). The location code for each sample is listed on figures and tables in the site-specific work plan. Existing well numbers are used when unique.

New wells will be designated by a SWMU or site number, followed by "MW" for permanent monitoring well, "TW" for temporary well, etc.; a "T" for Tetra Tech NUS; and numbered sequentially, by SWMU, beginning with zero one (MWT01).

Well clusters that are newly installed by TtNUS (two or more wells in close proximity) in the same investigative effort will be designated with the letter P followed by the number 2, 3, 4, etc. The number signifies the well depth in relation to other wells in that cluster. This two-digit code will follow the well identification. Using well T02 as an example of a three-well cluster, the well identification will be as follows:

T02	=	The deepest well in the cluster.
T02P2	=	Intermediate well
T02P3	=	Shallowest well in the cluster

Note: To keep the sample identification nomenclature to a minimum number of characters and to avoid redundancy, "MW" (monitoring well) is used for text, figures, and tables and replaced with "GW" (groundwater) in the sample identification. For example, MWT01 would be GWT01.

Round Identifier and/or Filtered:

This code section will be used for aqueous samples only.

Round Identifier:

A two-digit round identifier will be used to track the number of aqueous samples (GW, SW) taken from a particular aqueous sample location. The first sample collected from a location will be assigned round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Filtered:

Water samples that are field filtered (dissolved analysis) will be identified with an "F" in the last code section. No entry in this segment signifies an unfiltered (total) sample.

Depth Interval:

This code section will be used for soil and sediment samples only.

The depth code is used to note the depth below ground surface (bgs) at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and

fourth specify the bottom interval in feet bgs (soil) or inches bgs (sediment) of the sample. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc.

Depth (for soils, in feet bgs)

0002 = soil collected from 0 to 2 feet bgs

0204 = soil collected from 2 to 4 feet bgs

0810 = soil collected from 8 to 10 feet bgs

Depth (for sediments, in inches bgs)

0006= sediment collected from 0 to 6 inches bgs

0612= sediment collected from 6 to 12 inches bgs

3.1.1 Examples of Sample Nomenclature

The first groundwater sample collected from existing monitoring well 01 at the Cast High Explosives Fill/B146 Incinerator (SWMU 16) for a filtered sample would be designated as 16GW0101F.

The second groundwater sample collected from existing monitoring well C20P2 at SWMU 16 for an unfiltered sample would be designated as 16GWC20P202.

The first unfiltered groundwater sample collected from new monitoring well MWT01 at SWMU 13 would be designated as 13GWT0101.

The second surface water sample collected from point 01 at SWMU 13 for an unfiltered sample would be designated as 13SW0102.

A surface soil sample collected from soil boring 03 at SWMU 16 at the 0- to 2-foot interval would be designated as 16SS030002.

A subsurface soil sample from the same soil boring 03 at an interval of 4 to 5 feet bgs would be designated as 16SB030405.

A sediment sample collected at SWMU 19 from 0 to 6 inches at location 14 would be designated as 19SD140006.

3.2 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the approved field sampling plan and QAPP. They will be designated using a different coding system. The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank
RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
AB = Ambient Conditions Blank
SB = Source Water Blank

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.2.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered ground water sample collected on June 3, 2000 would be designated as FD06030001F.

The third duplicate of the day taken of a subsurface soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

The only rinsate blank collected on November 17, 2001 would be designated as RB11170101.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-03

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, and labels are required.

Site logbook
Field logbook
Sample label
Chain-of-Custody Form
Custody seals
Equipment calibration log
Monitoring Well Inspection Form
Water-Level Measurement Form
Low-Flow Purge Data Sheet
Ground Water Sample Log Sheet
Surface Water Sample Log Sheet

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

3.1 Site Logbook

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the field operations leader (FOL).

3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL must include the name of the laboratory in the "Remarks" section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page __ of __" on each COC. The original (top) signed copy of the COC will be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Task Order Manager (TOM). The COC Form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

3.5 Custody Seal

The custody seal is an adhesive-backed label with a number on each seal. It is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the TOM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment (e.g., multi-parameter water-quality meter) used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.7 Monitoring Well Inspection Form

The Monitoring Well Inspection Form is used to document the inspection of existing monitoring wells in accordance with SOP CTO166-09.

3.8 Water-Level Measurement Form

The Water-Level Measurement Form is used to document the determination of water levels in monitoring wells in accordance with SOP CTO166-18.

3.9 Low-Flow Purge Data Sheet

The Low-Flow Purge Data Sheet is used to document field measurements made while purging wells to stabilization in accordance with SOP CTO166-16.

3.10 Ground Water Sample Log Sheet

The Ground Water Sample Log Sheet is used to document the samples taken from a monitoring well at the end of low-flow well purging. This sheet is used in conjunction with SOP CTO166-05.

3.11 Surface Water Sample Log Sheet

The Surface Water Sample Log Sheet is used to document the samples collected from surface waters. This sheet is used in conjunction with SOP CTO166-19.

3.12 Soil and Sediment Sample Log Sheet

The Soil and Sediment Sample Log Sheets are used to document the sampling of soils and sediments. This sheet is used in conjunction with SOP CTO166-08 and SOP CTO166-20.

4.0 ATTACHMENTS

1. Chain-of-Custody Record

ATTACHMENT 1

CHAIN-OF-CUSTODY RECORD

TETRA TECH NUS, INC. CHAIN OF CUSTODY | NUMBER | PAGE ____ OF _____

PROJECT NO:	SITE NAME:	PROJECT MANAGER AND PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER AND PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER	CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE
PLASTIC (P) or GLASS (G)

PRESERVATIVE
USED

TYPE OF ANALYSIS

DATE YEAR ___	TIME	SAMPLE ID	MATRIX	GRAB (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

3/99
FORM NO. TtNUS-001

CHAIN-OF-CUSTODY RECORD

STANDARD OPERATING PROCEDURE
NUMBER CTO166-04

BOREHOLE ADVANCEMENT AND SOIL CORING USING DIRECT-PUSH TECHNOLOGY

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using direct-push technology (DPT) at the NSWC Crane facility. For this investigation, a Geoprobe® rig with a Macrocore Sampler will be the type of DPT used.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Cut-resistant non-latex Impermeable Gloves
Cotton gloves
Disposable medical-grade gloves (e.g., latex, nitrile)
Writing utensil
Boring log sheets: A copy of this form is included in SOP CTO166-07
Photoionization detector (PID) (see SOP CTO 166-06)
Geoprobe® or equivalent DPT equipment
Geoprobe® Macrocore Sampler or equivalent
Geoprobe® Sampling Kit or equivalent
Clear acetate liners: one new liner for each soil core
Required decontamination materials (see SOP CTO166-17)
Bentonite pellets

3.0 BOREHOLE ADVANCEMENT AND SOIL SAMPLING USING A GEOPROBE®

DPT will be employed to collect soil cores. DPT refers to sampling tools and sensors that are driven directly into the ground without the use of conventional rotary drilling equipment. DPT typically utilizes hydraulic pressure and/or percussion hammers to advance the sampling tools. Geoprobe® is a

manufacturer of a hydraulically powered, percussion/probing machine utilizing DPT to collect subsurface environmental samples.

3.1 Clear the area to be sampled of any surface debris (herbaceous vegetation, twigs, rocks, litter, etc.).

3.2 Place a new clear acetate liner in the detachable Macrocore core barrel and attach the coring device to the Geoprobe® rig.

3.3 Drive the macrocore sampler (lined with acetate) into the ground to a depth of 2 feet using hydraulic pressure. The 0- to 2-foot depth soil interval is considered to be the surface soil.

3.4 Retract the sampler from the borehole and remove the acetate liner and the soil core from the Macrocore barrel.

3.5 Attach the metal trough from the Geoprobe® Sampling Kit firmly to the tailgate of a vehicle. If a vehicle with a tailgate is not available, secure the trough on another suitable surface.

3.6 Place the acetate liner containing the soil core in the trough.

3.7 While wearing cut-resistant gloves (constructed of non-latex over cotton), cut the acetate liner through its entire length using the double-bladed knife that accompanies the Geoprobe® Sampling Kit. Then remove the strip of acetate from the trough to gain access to the collected soils. **CAUTION:** Do not attempt to cut the acetate liner while holding it in your hand.

3.8 Scan the entire length of the soil core for VOCs using the PID. Record the specific depth interval and the associated PID reading on the Boring Log Sheet. Collect a soil VOC sample using Encore samplers from the soil interval that had the highest PID reading. If no above-background PID readings were detected, collect the VOC sample from an interval that is discolored or displays other visual signs of being contaminated. If no visual sign of contamination is evident, collect the soil VOC sample from the center of the core interval (i.e., 1-foot depth). Details for collecting a VOC sample using the Encore sampler are included in SOP CTO166-08.

3.9 Log the soil core on the Boring Log Sheet (see SOP CTO166-07).

3.10 Place the soil core in a stainless-steel mixing bowl, homogenize, and collect the remainder of the soil sample aliquots, as described in SOP CTO 166-8.

3.11 Repeat steps 3.2 through 3.11 for the next depth intervals.

3.12 The depth to bedrock should be recorded on the Boring Log and the estimated moisture content of the soil and the presence or absence of water in the boring should be noted.

3.13 If readings from the PID are all at background levels below field screening criteria, then excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole.

3.14 If screening instruments indicate that contaminants may be present in the soil materials, then all excess soil core materials will be placed in a plastic bag (or drum if larger quantities). The bag will be tagged identifying the locations and depths from where the soils came from and the date. The bag will then be placed in a 55-gallon drum and stored on site until laboratory analyses of the soil are completed and classification of the soil waste materials can be determined (see SOP CTO166-15).

3.15 If soil materials from the boring are suspected of being contaminated (see 3.14 above), the soil boring will be backfilled with bentonite pellets up to the ground surface.

3.16 Decontaminate all soil sampling equipment in accordance with SOP CTO166-17 before collecting the next sample.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-05

MONITORING WELL SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for monitoring well sampling. Low-flow sampling techniques will be used for ground water sampling at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow sampling of monitoring wells:

Ground Water Sample Log Form: A copy of this form is attached at the end of this SOP

Bound field log book

Chain-of-Custody Form

Bladder pump

Surgical gloves

Labeled sample containers: Sample containers are certified clean by the laboratory supplying the sample containers.

Tag for each sample container

Plastic storage bags

Shipping containers with ice

3.0 MONITORING WELL SAMPLING PROCEDURES

3.1 Ground water sampling may be initiated when the monitoring well has been purged and stabilized in accordance with SOP CTO166-16.

3.2 Record the sample start time (using military time) on the Ground Water Sample Log Sheet. Record the field measurements for pH, (ORP), specific conductance, temperature, dissolved oxygen, and turbidity.

3.3 With the pump continuing to run, disconnect the flow-through cell from the pump discharge tube and immediately start filling sample bottles directly from the pump discharge. All sample containers will be supplied by the laboratory, and the laboratory will pre-preserve all sample containers, where appropriate.

3.4 Allow the pump discharge to flow gently down the inside of the container with minimal turbulence when filling sample containers. Avoid immersing the discharge tube into the sample as the sample container is being filled. Sample containers for volatile constituents (VOCs) must be completely filled so that no headspace exists in the container. The VOC vials will be filled to the top so that a convex meniscus is formed. Gently secure the cap, turn the vial upside down, and check to see if any air has been trapped inside the vial. If so, open the cap, reform the meniscus, and attempt again to secure the lid without trapping air in the sample. All other sample containers can have air space included when the container lid is secured.

3.5 Cap each container immediately after filling.

3.6 Record the sample time on the Ground Water Sample Log Form, the sample tag, and the sample label.

3.7 Secure the associated tag to each sample container.

3.8 Place the tagged sample container into a plastic storage bag and then into a cooler containing ice.

3.9 Enter the proper information on the Chain-of-Custody Form for each sample container (see SOP CTO166-03).

3.10 Repeat steps 3.3 through 3.9 for each sample container collected.

3.11 The pump rate should not be adjusted after sampling has commenced. If it becomes necessary to adjust the pump rate, document the change on the Ground Water Sample Log Form.

3.12 All samples will be collected into pre-preserved bottles (if required) supplied by an approved laboratory. All samples will be collected in the following sequence (where applicable):

- Volatile organic compounds (VOCs)
- Other organics
- Appendix IX Metals plus Sn (totals)
- Nitrate
- Nitrite

3.13 If the last turbidity measurement prior to the commencement of sampling showed turbidity to be greater than 10 nephelometric turbidity units (NTUs), then filtered aliquots of ground water will be collected and analyzed for dissolved metals. Without turning off the pump, attach a disposable, inline, 0.45-um filter cartridge at the end of the discharge tube. Fill sample containers marked for dissolved metals so that the laboratory knows that these aliquots are distinct sample fractions and that the results should be reported as dissolved analytes.

3.14 Repeat steps 3.5 through 3.9 for the filtered sample containers.

3.15 After completion of sample collection, remove the bladder pump from the well and decontaminate following the procedures in SOP CTO166-17.

3.16 Replace the outer protective well cap and lock the well.

3.17 All equipment should be cleaned and packed into the sample vehicle, along with the sample cooler for transport. Disposable gloves and other equipment should be placed in a plastic trash bag and handled as investigation-derived waste (SOP CTO166-15).

4.0 ATTACHMENTS

1. Ground Water Sample Log Sheet

ATTACHMENT 1

GROUND WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

GROUNDWATER SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Domestic Well Data
[] Monitoring Well Data
[] Other Well Type: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____
Type of Sample:
[] Low Concentration
[] High Concentration

SAMPLING DATA:

Date:	Color	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Time:	(Visual)	(S.U.)	(mS/cm)	(°C)	(NTU)	(mg/l)	(%)	
Method:								

PURGE DATA:

Date:	Volume	pH	S.C.	Temp.	Turbidity	DO	Salinity	Other
Method:								
Monitor Reading (ppm):								
Well Casing Diameter & Material								
Type:								
Total Well Depth (TD):								
Static Water Level (WL):								
One Casing Volume(gal/L):								
Start Purge (hrs):								
End Purge (hrs):								
Total Purge Time (min):								
Total Vol. Purged (gal/L):								

SAMPLE COLLECTION INFORMATION:

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
NUMBER CTO166-06

CALIBRATION AND USE OF PHOTOIONIZATION DETECTOR

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes procedures for the maintenance, calibration, and use of a photoionization detector (PID). The Photovac 2020 Photoionization Air Monitor will be used during the NSWC Crane facility investigation. The procedures for its use are discussed in detail in the following sections.

2.0 GLOSSARY

Electron-volt (eV) - A unit of energy equal to the energy acquired by an electron when it passes through a potential difference of 1 volt in a vacuum.

Intrinsically Safe (I.S.) - Based on wiring, configuration, design, operation, gasketing, construction, this instrument may be employed within locations where flammable gases and/or vapors may exist.

Ionization Potential (I.P.) - The energy required to remove an electron from a molecule yielding a positively charged ion and a negatively charged free electron. The instrument measures this energy level.

Photoionization Detector (PID) - Photoionization detector is employed as the general reference to air monitors of this type. PID's detection method employs ultraviolet (UV) radiation as an energy source. As air and contaminants are drawn through the ionization chamber, the UV light source causes the contaminant with ionization potentials equal to or less than the UV source to break into positive and negatively charge ions. The created ions are subjected to an electrostatic field. The voltage difference is measured in proportion to the calibration reference and the concentration of the contaminant.

Ultraviolet Radiation (UV) Lamp - Ultraviolet radiation is the energy source employed by the instrument to ionize collected sample gas streams. The UV lamp source is required to be equal to or greater than the ionization potential of the substance drawn through the instrument in order to create separate ionized species.

3.0 REQUIRED EQUIPMENT

Pen
Equipment Calibration Form
Photoionization detector
Isobutylene calibration gas (i.e., span gas)
Regulator

4.0 PROCEDURES

4.1 Principle of Operation

The Photovac portable photoionizer detects many organic (and a few inorganic) species. The basis for detection of this instrument is the ionization of components in gaseous streams. The incoming gas molecules are subjected to ultraviolet (UV) radiation, which is energetic enough to ionize many compounds associated with industrial activities. Molecules are transformed into charged-ion pairs, creating a current between two electrodes. Each molecule has a characteristic ionization potential, which is the energy required to remove an electron from the molecule, yielding a positively charged ion and a free electron. The instrument measures this energy level.

This instrument measures the concentration of airborne photoionizable gases and vapors and automatically displays and records these concentrations. It does not distinguish between individual substances. Readings displayed represent the total concentration of all photoionizable chemicals present in the sample. This instrument is factory-set to display concentration in units of ppm or mg/m^3. The meter display updates itself once per second.

The 2020 also performs short-term exposure limit (STEL), time-weighted average (TWA), and PEAK calculations. Any of these results can be viewed, but only one mode may be viewed at a time.

The 2020 has six keys for alphanumeric entry and for accessing multiple functions. The keys are used to set up and calibrate 2020. They allow the user to manipulate the concentration data in various ways.

All information entered with the keys and stored in the 2020's memory is retained when the instrument is switched off. The clock and calendar continue to operate and do not need to be set each time the 2020 is turned on.

4.1.1 Displays

The 2020 has a meter display for reporting detected concentration and a display used for status/ information to guide you through configuration options. All functions of the 2020 will be controlled or reported using one of these displays.

4.1.1.1 Meter Display

The meter display is four digits. It will always be used for reporting detected concentration. When the detector and pump are off, the meter display will be blank.

In order to accommodate the range of concentrations the 2020 can detect, the meter reading will be reported using one of two resolutions. A resolution of 0.1 will be used for concentrations below 100 ppm, and a resolution of 1 will be used for concentrations above 100 ppm.

4.1.1.2 Status Display

The status display is a two- line by 16-character display. The top line is used to display status information and prompts the user for inputs. The bottom line is used for soft key names. Up to three names can be displayed for the three soft keys. If a name does not appear for a soft key, then the soft key has no associated function.

4.1.2 Keys

4.1.2.1 Fixed Keys

The three round keys below the soft keys each have a fixed function. The first key is the ON/OFF key, the middle key is the EXIT key, and the last key is the ENTER key.

The ON/OFF key is used to both turn power on and off. To turn on the 2020, press the ON/OFF key. To turn the power off, press the ON/OFF key and hold it down for 2 seconds, and then release it. This is done to prevent accidental power off.

The EXIT key provides a way of returning to the default display. In the functional map, the soft keys allow the user to advance and the EXIT key provides a way to go back. If the user is at the initial entry of the menu, EXIT will return you to the default display.

The ENTER key has a context sensitive function. When the user is operating or navigating through the function map, the ENTER key is used to exit the functions and return to the default display. When entering data such as a name, number, date, or time, ENTER is used to confirm the entry.

4.1.2.2 Soft Keys

The three soft keys on the 2020 are located directly below the status display. Each key has varying functions for configuring the 2020, editing the data logger, and controlling the display. Because only three soft keys are available, each function is broken down into a path.

4.1.2.3 Entering Text with the Soft Keys

For all information that is entered, the left, center, and right soft keys correspond to the up, down, and right arrow.

The up and down arrows are used to change the character highlighted by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right most character, it wraps around to the first character again. To accept the changes, press the ENTER key. To ignore the change, press EXIT.

Formatting characters, such as the colon (:) in the time, the decimal (.) in a concentration, and the slash (/) in the date, are skipped when advancing the cursor.

All inputs are an eight-character input, which is displayed on the right side of the top line of the status display. The prompt, describing the input, occupies the left half of the top line. The soft keys are defined on the bottom line of the status display.

4.2 Default Display

The meter display shows the detected concentration. The resolution of the display changes with the magnitude of the reading. A reading of 0 to 99.9 will be displayed with a resolution of 0.1 ppm. A reading greater than 99.9 will be shown with a resolution of 1 ppm or 1 mg/m^3. The meter will display concentrations up to 2000 ppm or 2(99) mg/m^3.

The status display is used to display the instrument status, date, time, units, and active soft keys.

The default display provides the following information: instrument status, current detected concentration, time, date, and measurement units. The status display toggles between showing time and units and then the date.

When the display mode is MAX, the date and time correspond to the date and time the MAX concentration was recorded. In TWA mode, the time represents the number of hours and minutes during which the TWA has been accumulating. For PEAK and STEL monitoring, the date and time correspond to the current date and time.

4.3 Monitoring

4.3.1 Instrument Status

The instrument status is shown on the left of the first line of the status display and on the Table and Graph outputs. Each status has a priority assigned to it. If more than one status is in effect, then the status with the highest priority is displayed until the condition is corrected or until the option is turned off.

4.3.2 Alarms

While operating the instrument, any one of three alarm conditions can occur. To accurately identify the source of the alarm, each type of alarm has been given a unique status.

In addition to the status, the 2020 also has an audible alarm and a visual alarm LED. To conserve power, the 2020 alternates between these two alarm indicators, rather than operating both concurrently. Different alarms are identified by the frequency at which the 2020 alternates as follows: PEAK alarm, 5 times per second; STEL alarm, 2.5 times per second; and TWA alarm, 1.25 times per second.

The left soft key is used for acknowledging alarms, and is labeled "Ack." If no alarm exists, then the "Ack" key is not shown. To clear the alarm, press the "Ack" key. Once acknowledged, the alarm indicators are cleared. The alarm status will remain until the alarm condition clears.

The 2020 updates the peak concentration once every second. Following every update, the peak concentration is compared to the peak alarm level, and, if exceeded, an alarm is triggered.

If a 15-minute average concentration exceeds the selected STEL, a STEL alarm is generated.

The TWA alarm is generated when the current average concentration over an 8-hour period, since the TWA was last cleared, has exceeded the TWA exposure limit.

During calibration, all alarms are disabled. Once the calibration is complete, the alarms are re-enabled.

4.4 STEL, TWA, MAX, and PEAK Operation

The 2020's meter display can be configured to show one of four values: STEL, TWA, PEAK, and MAX.

4.4.1 Short-term Exposure Limit Mode

The STEL mode displays the concentration as a 15-minute moving average. The 2020 maintains 15 samples, each representing a 1-minute averaging interval.

Once every minute, the oldest of the 15 samples is replaced with a new 1-minute average. This moving average provides a 15-minute average of the last 15 minutes with a 1-minute update rate. Because the average is calculated using 15 one-minute averages, the meter display will only update once every minute.

The STEL is set to zero each time the instrument is turned on. Because STEL is a 15-minute moving average, there is no need to clear or reset the STEL.

STEL calculations are always being performed by the 2020. The user can display the results of the calculations by selecting "STEL" as the Display mode.

4.4.2 Time-weighted Average (TWA) Mode

The TWA accumulator sums concentrations every second until 8 hours of data have been combined. If this value exceeds the TWA alarm setting, a TWA alarm is generated. The TWA is not calculated using a moving average. Once 8 hours of data have been summed, the accumulation stops. In order to reset the TWA accumulator, press the "Clr" key.

This sum will only be complete after 8 hours, so the meter displays the current sum divided by 8 hours. While in TWA mode, the time on the status display will show the number of minutes and hours of data that TWA has accumulated. When this reaches 8 hours, the 2020 stops accumulating data and the TWA is complete.

TWA calculations are always being performed by the 2020. The user can display the results of the calculations by selecting TWA as the Display mode.

4.4.3 MAX Mode

The MAX mode displays the maximum signal, with the date and time that it was recorded. The 2020 continues to log data according to the selected averaging interval, but only the maximum detected concentration is displayed on the meter display.

The right soft key is used to clear the meter when displaying MAX. The "Clr" key only affects the reading that the meter is displaying. For example, if the MAX reading is displayed, and "Clr" is pressed, only the MAX value is cleared. The TWA is still accumulating in the background.

4.4.4 PEAK Mode

The PEAK mode displays the current detected concentration. The reading is updated once a second. In the background, the 2020 data logger is sampling the concentration and measuring minimum, maximum, and average concentrations for the selected averaging interval. At the end of every interval, one entry is placed in the data logger until the data logger is full. For CTO 166, the instrument should be operated in this mode. Operation within the other specialized modes is the responsibility of the SSO.

4.5 Set Functions

Set functions are used to set up the 2020. Three functions can be set on the 2020: Pump, Clock, and Calibration.

4.5.1 Pump

The Pump function is used to control the pump. After selecting "Set Pump," the 2020 responds by displaying the new pump status.

The detector is also turned off when the pump is turned off. This prevents the detector from being damaged when there is no sample flowing through the detector.

When the pump and the detector are off, the meter display will be blank. Turn the pump and detector off when concentration measurements are not necessary, and the 2020 will only be used for reviewing data

or generating reports. By operating the instrument with the pump and detector off when they are not needed, the lives of the battery and ultraviolet (UV) lamp will be conserved.

To set the pump:

1. Press the ENTER key. The top line of the status display changes to "Select?" The bottom line displays three soft key names: "Set," "Log," and "Disp."

2. Press the soft key below "Set."

3. The names of the soft keys change to reflect the Set options. The display now shows three devices that can be set: "Clock," "Pump," and "Cal." Press the "Pump" key.

4. The 2020 turns the pump off. If the pump was off, pressing "Pump" will turn the pump on.

5. A message will be displayed to show the status of the pump. The 2020 reverts back to the previous menu after a few seconds.

6. To return to the default display, press the ENTER key.

4.5.2 Clock

The Clock function is used to set both the current date and time.

To set the clock:

1. Press the ENTER key.

2. Press the "Set" key.

3. When the names of the soft keys change, press the "Clock" key.

   The up and down arrows are used to change the character underlined by the cursor. The right arrow is used to advance the cursor to the next character on the right. When the cursor is advanced past the right-most character, it wraps around to the first character again.

Formatting characters, such as the colon (:) in the time and the slash (/) in the date are skipped when advancing the cursor.

4. Use the "arrow keys" to enter the correct time. The time is formatted as Hour:Minute:Second.

5. Press the ENTER key to confirm the time and move to the date option.

6. When setting the date, the 2020 prompts for the current date formatted as Year/Month/Day. Use the "arrow keys" to enter the correct date.

7. Press the ENTER key to confirm the date and return to the Set options. Wait for the display to timeout or press ENTER to return to the default display.

4.5.3 Calibration (Cal)

"Cal" allows the user to set up and calibrate the 2020. There are three options under the Cal function: "Zero," "Span," and "Mem."

A calibration memory consists of a name, a response factor, and PEAK, TWA, and STEL alarm levels.

The "Zero" and "Span" keys are covered in detail in the manufacturer's operations manual for the instrument.

To edit the calibration memory, select "Mem" and then "Chng." The 2020 prompts the user with two new soft keys: "User" and "Lib."

4.5.4 Library (Lib)

Library selections simplify Cal Memory programming and provide standard response factors for approximately 70 applications. "Lib" allows the user to select an entry from a pre-programmed library. The name, response factor, and three alarm levels are all set from the library. To select a library entry to program the selected Cal Memory:

1. Select "Set," "Cal," "Mem," "Chng," and "Lib."
2. Use the "Next" and "Prev" keys to scroll through the list. See the manufacturer's manual Appendix 8.7 for a list of the library entries.

4.6 Preparing for Field Operation of the Photovac 2020

Turning The 2020 On

1. Turn the 2020 on by pressing the ON/OFF key.

2. The 2020 will display the software version number. Wait for the 2020 to proceed to the default display.

3. Allow 10 minutes for the instrument to warm up and stabilize.

4. Press the Enter Key. The default display will provide three soft key selections, "Set," "Log," and "Display."

5. Press "Set." From this option, three other soft key selections will be offered: "Pump," "Clock," and "Cal."

6. Press "Cal." This will begin the calibration sequence. The first selection is to zero the instrument.

7. Press Enter; zeroing will begin. (Note: When employing zero gas, attach and activate zero gas supply at this time.)

8. The next selection offered will be Span. Press Enter, at which time the concentration will be requested. The isobutylene calibration gas employed under general service will be marked on the side of the container. Use the soft keys to toggle into position and to log the concentration. Once the concentration is logged, press "Enter." The direction on the status display will indicate spanning. At this time, hook up the span gas with a regulator to the Photovac 2020 and open it to supply enough flow to elevate the flow rate indicator to the green indicator line (1/8 inch from the rest position).

9. Once spanning is complete, the alarms, which have been disabled during calibration, will activate, indicating that calibration is complete.

10. Document this calibration procedure using a Document of Calibration form (included in Appendix A).

This instrument is ready for general purpose application.

Calibration is to be performed daily or prior to each use, in accordance with this section.

4.7 Maintenance and Calibration Schedule

Function	Frequency
Routine Calibration	Prior to each use.
Factory Inspection and Calibration	Once a year, or when malfunctioning.
Wipe Down the Outer Casing of the Unit	After each use.
Clean UV Light Source	Every 24 hours of operation.
Sample Inlet Filter	Change on a weekly basis or as required by level of use.
Battery charging	After each use.
Clean ionization chamber	Monthly.

4.7.1 Cleaning the UV Light Source Window

1. Turn the FUNCTION switch to the OFF position. Use the 2020 multi-tool and remove the lamp housing cover. CAUTION: The UV lamp is delicate and expensive; handle carefully.

2. Tilt the lamp housing with one hand over the opening; slide the lamp out of the housing.

3. The lamp window may now be cleaned with any of the following compounds using lens paper:
   a. 11.7 eV Lamp - Dry Aluminum Oxide Powder (3.0 micron powder)
   b. All other lamps-HPLC Grade Methanol

4. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace the o-ring as necessary, reinstall the lamp housing cover, and tighten it using the 2020 multi-tool. (Do not over tighten.)

5. Recalibrate the instrument as per Section 4.6.

4.7.2 Cleaning the Ionization Chamber

1. Turn the FUNCTION switch to the OFF position and remove the lamp housing cover and lamp as per Section 4.7.1.

2. Using a gentle jet of compressed air, gently blow out any dust or dirt.

3. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Replace the o-ring as necessary, reinstall the lamp housing cover, and tighten it using the 2020 multi-tool. (Do not over tighten.)

4. Recalibrate the instrument as per Section 4.6.

4.8 Instrument Advantages

The Photovac 2020 is easy to use in comparison to many other types of monitoring instrumentation. Its detection limit range is in the low parts-per-million range. Response rapidly reaches 90 percent scale of the indicated concentration (less than 3 seconds for benzene). This instrument's automated performance covers multiple monitoring functions simultaneously, incorporating data logging capabilities.

4.9 Limitations of the Photovac 2020 Photoionization Monitor

- Because the 2020 is a nonspecific total gas/vapor detector, it cannot be used to identify unknown chemicals; it can only quantitate them in relationship to a calibration standard (relative response ratio).

- For appropriate application of the 2020, ionization potentials of suspected contaminants must be known.

- Because the types of compounds that the 2020 can potentially detect are only a fraction of the chemicals possibly present at a hazardous waste site or incident, a background or zero reading on this instrument does not necessarily signify the absence of air contaminants.

- The 2020 instrument can only monitor certain vapors and gases in air. Many nonvolatile liquids, toxic solids, particulates, and other toxic gases and vapors cannot be detected.

- PIDs are generally not specific. Their response to different compounds is relative to the calibration gas used. This is referred to as relative response ratio. Instrument readings may be higher or lower than the true concentration. This can be an especially serious problem when monitoring for total contaminant concentrations if several different compounds are being detected at once.

- The 2020 is a small, portable instrument that cannot be expected to yield results as accurately as laboratory instruments.

4.9.1 Variables Affecting Monitoring Data

Monitoring a hazardous waste site environment can pose a significant challenge in assessing airborne concentrations and the potential threats to site personnel. Several variables may influence both dispersion and the instrument's ability to detect actual concentrations. Some of the variables, which may impact these conditions, are as follows:

- Temperature - Changes in temperature or pressure will influence volatization and affect airborne concentrations. Additionally, an increase or decrease in temperature ranges may have an adverse effect on the instrument's ability to detect airborne concentrations.

- Humidity - Excessive levels of humidity may interfere with the accuracy of monitoring results.

- Rainfall - Through increased barometric pressure and water, rainfall may influence dispersion pathways affecting airborne emissions.

- Electromagnetic interference - High voltage sources, generators, other electrical equipment may interfere with the operation and accuracy of direct-reading monitoring instruments.

5.0 TROUBLESHOOTING

5.1 <u>Fault Messages</u>

When the "Fault" status is displayed, the 2020's operation is compromised.

Fault 1: Signal from zero gas is too high.

Cause: If another fault occurred while the 2020 was setting its zero point, then this fault is displayed.
Action: Ensure no faults are occurring and calibrate the 2020 again.

Cause: Contamination of sample line, sample probe, or fittings before the detector.

Action: Clean or replace the sample line, sample probe, or the inlet filter.

Cause: Span gas and zero air are switched.

Action: Ensure clean air is used to zero the 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Cause: Ambient air is contaminated.

Action: If you are unsure about the quality of ambient air, use a supply of commercial zero grade air to zero the 2020.

Fault 2: Signal from span gas is too small.

Cause: Operator may have switched the span gas and zero air.

Action: Ensure clean air is used to zero the 2020. If you are using gas bags, mark the calibration and zero gas bags clearly.

Action: Ensure the span gas is of a reliable concentration.

Cause: UV lamp window is dirty.

Note: Do not remove the detector lamp in a hazardous location.

Action: Clean the UV lamp window.

Cause: UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Incompatible application.

Action: The concentration and sample gas are incompatible for use with the 2020.

Fault 3: UV lamp fault. UV lamp has not started.

Cause: The UV lamp has not started immediately.

Action: This fault may be seen momentarily when 2020 is first turned on. Allow 30 to 60 seconds for the UV lamp to start and the fault to clear.

Cause: The UV lamp serial number label is blocking the photocell.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the label is blocking the photocell. Rotate the lamp approximately 90 degrees and then try to start the 2020 again. If the fault persists, replace the lamp.

Cause: the UV lamp is not installed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a UV lamp.

Cause: The UV lamp has failed.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: Install a new UV lamp.

Cause: Electronic problem.

Action: If a new UV lamp still generates this fault, then contact the Photovac Service Department.

Fault 4: Pump current too low or too high.

Cause: If the pump sounds labored, then the pump is operating beyond normal operating parameters.

Action: Check for an obstruction in the sample line. Make sure sample line, sample probe, or inlet filter are not plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Action: Ensure the sample outlet, located on the underside of the 2020, is not obstructed.

Cause: The UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the TtNUS equipment manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the TtNUS equipment manager.

Cause: The pump has failed.

Action: Contact the TtNUS equipment manager.

5.2 Specific Problems

Problem: Very low or no instrument response detected, yet compounds are known to be present.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 4.6 of the User's Manual. After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the TtNUS equipment manager.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Disconnect the battery charger before calibrating the 2020.

Cause: Calibration memories have not been programmed correctly.

Action: Program all the calibration memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory.

Cause: The response factor has been set to zero.

Action: Enter the correct response factor. Refer to Appendix 8.6 for a list of response factors. If the compound is not listed in Appendix 8.6 or you are measuring gas mixtures, then enter a value of 1.0. See User's Manual provided by the manufacturer.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: Detector is leaking. A decrease in sensitivity may be due to a leak in the detector.

Note: Do not remove or replace the detection lamp in a hazardous location.

Action: Ensure the UV lamp has been installed correctly.

Action: Ensure the lamp cover has been tightened down. Do not overtighten the cover.

Action: Ensure the o-ring seal on the lamp cover is positioned correctly.

Cause: The UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the TtNUS equipment manager.

Cause: UV lamp is too wide, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the TtNUS equipment manager.

Cause: The sampling environment is extremely humid.

Action: Water vapor is not ionized by the PID, but it does scatter and absorb the light and results in a lower reading. The 2020 detector has been designed to operate under high humidity conditions. Under extreme conditions, you may notice decreased response due to humidity.

Cause: The UV lamp is failing.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: High concentration of non-ionizable compounds. Chemical compounds, such as methane, with IPs greater than the 10.6 eV scatter and absorb the UV light. Sensitivity may be decreased significantly. Application with high backgrounds of such materials may be incompatible with the 2020. Contact the Photovac Applications Group for more information.

Problem: Erroneously high readings.

Cause: Sampling environment is extremely humid.

Action: Water vapor may contain mineral salts, which carry a charge. The water vapor becomes an electrolytic solution, which becomes ionized when it enters the detector. Atmospheric water in areas around the sea or stagnant water may produce a response in the absence of contaminants. The same effect may be seen when conducting ground water investigations in areas where the water is hard because it contains a significant concentration of minerals.

Cause: The 2020 has not been calibrated properly.

Action: Ensure the calibration gas is of a reliable concentration and then calibrate the instrument as outlined in Section 4.6. After the instrument has been calibrated, sample the bag of calibration gas. A reading equivalent to the calibration gas should be displayed. If not, contact the TtNUS equipment manager.

Cause: Cal Memories have not been programmed correctly.

Action: Program all the Cal Memories you require for your application. You must use the correct calibration gas and concentration for each Cal Memory. See Section 3.4 of the User's Manual.

Cause: You are not using the correct Cal Memory.

Action: Select the correct Cal Memory for your application. See Section 3.2.2 or 3.3.2 of the User's Manual.

Note: It does not matter which Cal Memory is selected or which response factor is entered. The 2020's response is not specific to any one compound. The reading displayed represents the total concentration of all ionizable compounds in the sample.

Cause: The detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the TtNUS equipment manager.

Problem: Date and time settings are not retained.

Cause: The battery pack was removed before the 2020 was turned off.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack and reset the time and date. Ensure that the 2020 has been turned off before removing the battery pack.

Cause: The 2020 has not been used for 3 months or more and the internal battery (not the external battery pack) has discharged.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Connect the 2020 to the AC adapter and turn the 2020 on. Turn the pump off. While the 2020 is running, the internal battery is charging. Leave the instrument running for approximately 24 hours.

Problem: Instrument status shows "Over."

Cause: High concentrations of gases and vapors will cause a rapid change in signal level. The detector and associated electronics may become temporarily saturated.

Action: Wait a few seconds for the status to return to normal. PIDs are designed to detect relatively low concentrations of gases and vapors. Exposure to very high concentrations may result in a very high or maximum response.

Cause: The detector has become saturated.

Action: Move the 2020 to a location where it can sample clean air. Sample clean air until the reading stabilizes around 0.

Cause: Detector has been short circuited by foreign matter in the detector cell.

Note: Do not service the 2020 in a hazardous location.

Action: Do not touch the wire grid inside the detector cell. Use a gentle jet of compressed air to remove any dust or dirt in the detector cell.

Warning: Do not insert any object, other than the UV lamp, into the lampholder.

Cause: There is an undetermined problem.

Action: Contact the TtNUS equipment manager.

Problem: Display is blank.

Cause: Battery pack is critically low.

Note: Do not remove or recharge the battery pack in a hazardous location.

Action: Replace the battery pack or connect the 2020 to the AC adapter.

Cause: The battery pack is not connected to the instrument correctly.

Action: Ensure the battery pack connector is securely attached to the connector on the 2020.

Cause: There is an undetermined problem.

Action: Reset the 2020. You must leave the instrument on while you disconnect the battery pack. This will reset the instrument. Reconnect the battery pack and close the battery hatch. Turn on the 2020, set the time and date, and program all the calibration memories that you are using.

Action: Contact the TtNUS equipment manager.

Problem: Sample flow rate is less than 300 ml/min.

Cause: The inlet filter is plugged.

Note: Do not replace the inlet filter in a hazardous location.

Action: Replace the inlet filter.

Cause: The inlet filter has not been installed properly.

Action: Ensure that the inlet filter has been installed correctly.

Cause: The UV lamp is too long, causing flow to be restricted.

Note: Do not remove or replace the detector lamp in a hazardous location.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too long for the lampholder. Replace the lamp and contact the TtNUS equipment manager.

Cause: The UV lamp is too wide, causing flow to be restricted.

Action: If you have a UV lamp with a white serial number label, it is possible that the lamp is too wide for the lampholder. Contact the TtNUS Equipment Manager.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter and liquid has been aspirated.

Action: Contact the TtNUS equipment manager.

Cause: Sample outlet is obstructed.

Action: Ensure the sample outlet is not obstructed in any way.

Cause: Pump has been damaged.

Action: Contact the TtNUS equipment manager.

Problem: Liquid has been aspirated.

Cause: The 2020 has been exposed to a solvent that can pass through the inlet filter.

Action: Contact the TtNUS equipment manager.

Problem: Corrosive gases and vapors have been sampled.

Cause: The 2020 has been exposed to corrosive gases and vapors.

Action: Corrosive gases and vapors can affect the electrodes within the detector as well as the lamp window. Prolonged exposure to corrosive materials may result in permanent fogging or etching

of the window. If the 2020 is exposed to corrosive material, contact the TtNUS equipment manager.

6.0 SHIPPING

The Photovac may be shipped as cargo or carried on as luggage if there is no calibration gas cylinder accompanying the kit. When shipping or transporting the calibration gas, a Hazardous Airbill must be completed.

7.0 REFERENCES

Photovac 2020 Photoionization Monitor User's Manual, 1995.

8.0 ATTACHMENTS

1. Equipment Calibration Log

ATTACHMENT 1

EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

EQUIPMENT CALIBRATION LOG

STANDARD OPERATING PROCEDURE
NUMBER CTO166-07

BOREHOLE AND SOIL SAMPLE LOGGING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the standard procedures and technical guidance on the logging of soil cores collected at the NSWC Crane facility.

2.0 FIELD FORMS AND EQUIPMENT

Knife
Ruler (marked in tenths and hundredths of feet)
Boring Log: An example of this form is attached.
Photoionization detector (PID) (see SOP 166-06)
Writing utensil

3.0 RESPONSIBILITIES

A field geologist or engineer is responsible for supervising all boring activities and assuring that each borehole is properly and completely logged.

4.0 PROCEDURES FOR BOREHOLE AND SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (attached to this SOP).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no distinguishable size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils will be divided into categories: rock fragments, sand, or gravel. The terms "sand" and "gravel" not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term "rock fragments" will be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges that are typically observed indicate little or no transport from their source area; and therefore, the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used, it will be followed by a size designation such as "(1/4 inchΦ-1/2 inchΦ)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

4.2 Color

Soil colors will be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Because color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples will be broken or split vertically to describe colors. Samplers tend to smear the sample surface, creating color variations between the sample interior and exterior.

The term "mottled" will be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

4.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are noncohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in the following table.

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist.
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb.
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort.
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort.
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail.
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample will be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods will be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

4.4 Weight Percentages

In nature, soils are consist of particles of varying size and shape and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., sandy)	31 - 50 percent

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

4.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the gloved hand or on a porous surface liberates water (i.e., dirties or muddies the surface). Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire field activity.

4.6 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table will be used.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Clay	CL	inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays,.
	CH	inorganic clays of high plasticity, fat clays.
	OH	organic clays of medium to high plasticity, organic silts.
Silt	ML	inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity.
	OL	organic silts and organic silty clays of low plasticity..
	MH	inorganic silts, micaceous or diatomaceous fine sand or silty soils.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Sand	SW	well graded sands, gravelly sands, little or no fines.
	SP	poorly graded sands, gravelly sands, little or no fines.
	SM	silty sands, sand-silt mixtures.
	SC	clayey sands, sand-clay mixtures.

4.7 Summary of Soil Classification

In summary, soils will be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (optional)
- Soil types
- Moisture content
- Other distinguishing features
- Grain size
- Depositional environment

4.0 ATTACHMENTS

1. Figure 1 - Unified Soil Classification System
2. Boring Log

ATTACHMENT 1

FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

Unified Soil Classification System

Coarse Grained Soils (more than half of soil > No. 200 sieve)	Gravels (More than half of coarse fraction > no. 4 sieve size)	GW	Well graded gravels or gravel-sand mixtures, little or no fines
		GP	Poorly graded gravels or gravel-sand mixtures, little or no fines
		GM	Sandy gravels, gravel-sand-silt mixtures
		GC	Clayey gravels, gravel-sand-silt mixtures
	Sands (More than half of coarse fraction < no. 4 sieve size)	SW	Well graded sands or gravelly sands, little or no fines
		SP	Poorly graded sands or gravelly sands, little or no fines
		SM	Silty sands, sand-silt mixtures
		SC	Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity
Fine Grained Soils (more than half of soil < No. 200 sieve)	Silts and Clays LL = < 50	ML	Inorganic silts and very fine sands, rock flour, silty fine sands or clayey silts with slight plasticity
		CL	Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, lean clays
		OL	Organic silts and organic silty clays of low plasticity
	Silts and Clays LL = > 50	MH	Inorganic silts, micaceous or diatomaceous fine sandy or silty soils, elastic silts
		CH	Inorganic silts of high plasticity, fat clays
		OH	Organic clays of high plasticity, organic silty clays, organic silts
Highly Organic Soils		Pt	Peat and other highly organic soils

Grain Size Chart

Classification	Range of Grain Sizes	
	U.S. Standard Sieve Size	Grain Size In Millimeters
Boulders	Above 12"	Above 305
Cobbles	12" to 3"	305 to 76.2
Gravel	3" to No. 4	76.2 to 7.76
coarse	3" to 3/4"	76.2 to 4.76
fine	3/4"to No.4	19.1 to 4.76
Sand	No. 4 to No. 200	4.76 to 0.074
coarse	No.4 to No. 10	4.76 to 2.00
medium	No. 10 to No. 40	2.00 to 0.420
fine	No. 40 to No. 200	0.420 to 0.074
Silt and Clay	Below No. 200	Below 0.074

Relative Density (SPT)

SANDS AND GRAVELS	BLOWS/FOOT
VERY LOOSE	0 – 4
LOOSE	4 – 10
MEDIUM DENSE	10 – 30
DENSE	32 – 50
VERY DENSE	OVER 50

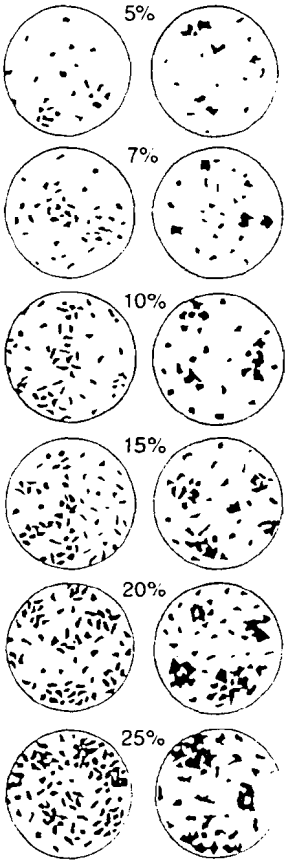
Consistency (SPT)

SILTS AND CLAYS	BLOWS/FOOT
VERY SOFT	0 – 2
SOFT	2 – 4
MEDIUM STIFF	4 – 8
STIFF	8 – 16
VERY STIFF	16 – 22
HARD	OVER 32

Group Symbols Chart

				Group Symbol		Group Name
GRAVEL % gravel % sand	5% fines	Well-graded		GW	<15% sand	Well-graded gravel
					15% sand	Well-graded gravel with sand
		Poorly-graded		GP	<15% sand	Poorly graded gravel
					15% sand	Poorly graded gravel with sand
	10% fines	Well-graded	fines = ML or MH	GW-GM	<15% sand	Well-graded gravel with silt
					15% sand	Well-graded gravel with silt and sand
			fines = CL or CH	GW-GC	<15% sand	Well-graded gravel with clay
					15% sand	Well-graded gravel with clay and sand
		Poorly-graded	fines = ML or MH	GP-GM	<15% sand	Poorly graded gravel with silt
					15% sand	Poorly graded gravel with silt and sand
			fines = CL or CH	GP-GC	<15% sand	Poorly graded gravel with clay
					15% sand	Poorly graded gravel with clay and sand
	15% fines		fines = ML or MH	GM	<15% sand	Silty gravel
					15% sand	Silty gravel with sand
			fines = CL or CH	GC	<15% sand	Clayey gravel
					15% sand	Clayey gravel with sand
SAND % sand % gravel	5% fines	Well-graded		SW	<15% gravel	Well-graded sand
					15% gravel	Well-graded sand with gravel
		Poorly-graded		SP	<15% gravel	Poorly graded sand
					15% gravel	Poorly graded sand with gravel
	10% fines	Well-graded	fines = ML or MH	SW-SM	<15% gravel	Well-graded sand with silt
					15% gravel	Well-graded sand with silt and gravel
			fines = CL or CH	SW-SC	<15% gravel	Well-graded sand with clay
					15% gravel	Well-graded sand with clay and gravel
		Poorly-graded	fines = ML or MH	SP-SM	<15% gravel	Poorly graded sand with silt
					15% gravel	Poorly graded sand with silt and gravel
			fines = CL or CH	SP-SC	<15% gravel	Poorly graded sand with clay
					15% gravel	Poorly graded sand with clay and gravel
	15% fines		fines = ML or MH	SM	<15% gravel	Silty sand
					15% gravel	Silty sand with gravel
			fines = CL or CH	SC	<15% gravel	Clayey sand
					15% gravel	Clayey sand with gravel

Group Symbol				Group Name
CL	<30% plus No. 200	<15% plus No. 200		Lean clay
		15-25% plus No. 200	% sand % of gravel	Lean clay with sand
			% sand <% of gravel	Lean clay with gravel
	30% plus No. 200	% sand % of gravel	<15% gravel	Sandy lean clay
			15% gravel	Sandy lean clay with gravel
		% sand <% of gravel	<15% sand	Gravelly lean clay
			15% sand	Gravelly lean clay with sand
ML	<30% plus No. 200	<15% plus No. 200		Silt
		15-25% plus No. 200	% sand % of gravel	Silt with sand
			% sand <% of gravel	Silt with gravel
	30% plus No. 200	% sand % of gravel	<15% gravel	Sandy Silt
			15% gravel	Sandy Silt with gravel
		% sand <% of gravel	<15% sand	Gravelly silt
			15% sand	Gravelly silt with sand
CH	<30% plus No. 200	<15% plus No. 200		Fat clay
		15-25% plus No. 200	% sand % of gravel	Fat clay with sand
			% sand <% of gravel	Fat clay with gravel
	30% plus No. 200	% sand % of gravel	<15% gravel	Sandy fat clay
			15% gravel	Sandy fat clay with gravel
		% sand <% of gravel	<15% sand	Gravelly fat clay
			15% sand	Gravelly fat clay with sand
MH	<30% plus No. 200	<15% plus No. 200		Elastic silt
		15-25% plus No. 200	% sand % of gravel	Elastic silt with sand
			% sand <% of gravel	Elastic silt with gravel
	30% plus No. 200	% sand % of gravel	<15% gravel	elastic silt
			15% gravel	elastic silt with gravel
		% sand <% of gravel	<15% sand	elastic silt
			15% sand	elastic silt with sand
OL/OH	<30% plus No. 200	<15% plus No. 200		Organic soil
		15-25% plus No. 200	% sand % of gravel	Organic soil with sand
			% sand <% of gravel	Organic soil with gravel
	30% plus No. 200	% sand % of gravel	<15% gravel	Sandy organic soil
			15% gravel	Sandy organic soil with gravel
		% sand <% of gravel	<15% sand	Gravelly organic soil
			15% sand	Gravelly organic soil with sand



ATTACHMENT 2
BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS*	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): [ ]

Remarks: _____

Connected to Well: Yes No Well I.D. #:

STANDARD OPERATING PROCEDURE
NUMBER CTO166-08

SURFACE AND SUBSURFACE SOIL SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for surface and subsurface soil sampling using direct-push technology (DPT) or split-barrel samplers during field activities at the NSWC Crane facility. This procedure also describes the collection of samples for analysis of volatile organic compounds (VOCs) using EnCore samplers and the use of field screening [i.e., photoionization detector (PID)] to select the most appropriate subsurface soil interval for VOC sampling.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil with indelible ink

Disposable medical-grade gloves (i.e. latex, nitrile)

Boring log

Soil sample logsheets

Stainless-steel mixing bowls

Stainless-steel trowel or soup spoon

EnCore handle and samplers

Required sample containers: All sample containers including shipping coolers for analysis by fix-based laboratories will be supplied and certified clean by the laboratory.

Required decontamination materials

Chain-of-custody records

Required personnel protective equipment (PPE)

Photoionization detector (PID) (see SOP 166-06)

Wooden stakes or pin flags

Sealable polyethylene bags

Heavy-duty cooler

Ice

Razor knife

Geoprobe® and sampling equipment

Sample labels and tags

3.0 COLLECTION OF SOIL SAMPLES FOR VOLATILE ORGANIC COMPOUNDS (VOCs)

When soil cores are collected using DPT such as Geoprobe®, 2-foot soil intervals will be collected in clear acetate tubes, which can be extracted from the Geoprobe® core barrel (see SOP CTO166-04) upon retrieval at the surface.

3.1 Slit the acetate liner lengthwise with a razor knife, remove a section of the liner, and expose the length of the soil interval (see SOP CTO166-04).

3.2 Scan the soil core interval with a PID, slowly moving the intake nozzle along the length of the core where the acetate liner has been slit open. Note on the boring log the range of PID readings that are detected and the specific location(s) along the sample interval where above-background readings are encountered. If elevated volatile organics are measured via the PID, collect the VOC samples from the specific interval where the highest PID reading is measured. If no above-background PID readings are measured, then the VOC sample will be collected from a specific interval where visual signs of contamination (staining, etc.) are observed. If no above-background PID reading is measured and no discoloration or odor in the soil core indicates potential contamination, then collect the VOC sample from near the center of a core.

3.3 The 0- to 2-foot core interval will be collected as a surface soil sample. Determine where in this core interval the highest PID reading was encountered. Soil samples collected for volatile organics will be obtained directly from soil cores using four EnCore samplers for each VOC sample. These samples are to be collected by pushing the EnCore samplers directly into the soil core where the highest PID readings were measured, ensuring that the sampler is packed tight with soil and leaving no headspace between cap and container. All four EnCore sample containers will be collected as close to each other as possible. Make sure that all caps are securely fastened to the samplers and locked in place with both clips (see instructions that come with samplers). Write the sample identification on the strip labels that come with the samplers and place a label on each of the four samplers.

3.4 Place the four EnCore samplers in the ziplock pouches that come with the samplers and fill in appropriate information, including sample identification, date, time, and other information on the label. Place the four pouches in a plastic bag and place the tag on the bag, identifying the sample identification and other necessary information (see SOP CTO166-01).

3.5 Once the samples are properly labeled, bagged, and tagged, place the sample into the cooler containing ice and a trip blank. The cooler should be kept at 4°C and shipped to the analytical laboratory for preservation or extraction within 48 hours.

3.6 Fill in the required information on the Soil Sample Log Sheet (attached at the end of this SOP) and fill in the required information on the Chain-of-Custody (COC) Form.

3.7 Up to two additional subsurface soil samples will be collected from each boring. For these subsurface soil samples, the collected cores will be scanned with the PID before a specific depth is selected for VOC sampling. Choose the specific location within the collected cores that had the highest PID reading and proceed to collect four EnCore samples, as described above in 3.2 through 3.6. If readings are not elevated, samples will be collected from the center of each cored interval using the default sample depths identified in the QAPP. Place the samples in the cooler containing ice, after they have been properly labeled, bagged, and tagged.

4.0 COLLECTION OF OTHER SOIL SAMPLE ALIQUOTS

Note: A surface soil sample is collected from the 0- to 2-foot depth (i.e., one core length). Additional subsurface soil samples each consist of two 2-foot core segments.

4.1 After the VOC sample has been collected for the soil interval of interest (see 3.0 above), the remainder of the soil interval will be composited and used to fill the remainder of the sample containers. Any surface debris (e.g., herbaceous vegetation, twigs, rocks, litter, etc.) should first be removed from the top of the surface soil core. For other core intervals, the top 2 inches of each core should be discarded because it often contains material scraped from the side of the borehole and not fresh material from the bottom of the borehole.

4.2 Slide the remaining core material out of the acetate liner and into a clean, decontaminated stainless-steel mixing bowl. Mix the soil thoroughly with a stainless-steel spoon and remove gravel, large pebbles, and other coarse materials. Fill the required sample containers in the following order:

- Containers for organic analyses
- Container for metals
- Container for Nitrate
- Container for Nitrite

4.3 Complete all required information on the sample labels (see SOP CTO166-01).

4.4 Fill in all required information on the sample tag and secure the tag to the sample container.

4.5 Place the sample container in a ziplock plastic bag and seal shut. Place the bag in a cooler containing ice and cool to 4 $\pm$ 2°C.

4.6 Record the required information on the Soil Sample Log Sheet and the COC Record form.

5.0 PACKAGING AND SHIPPING OF SAMPLES

Samples will be packaged and shipped according to SOP CTO166-04.

6.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet
2. Chain of Custody Record

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.Q.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

Date:	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

ATTACHMENT 2

CHAIN-OF-CUSTODY RECORD

TETRA TECH NUS, INC. CHAIN OF CUSTODY NUMBER PAGE ____ OF ____

PROJECT NO:	FACILITY:	PROJECT MANAGER	PHONE NUMBER	LABORATORY NAME AND CONTACT:
SAMPLERS (SIGNATURE)		FIELD OPERATIONS LEADER	PHONE NUMBER	ADDRESS
		CARRIER/WAYBILL NUMBER		CITY, STATE

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

CONTAINER TYPE
PLASTIC (P) or GLASS (G)

PRESERVATIVE
USED

TYPE OF ANALYSIS

DATE YEAR	TIME	SAMPLE ID	LOCATION ID	TOP DEPTH (FT)	BOTTOM DEPTH (FT)	MATRIX (GW, SO, SW, SD, QC, ETC.)	COLLECTION METHOD GRAP (G) COMP (C)	No. OF CONTAINERS									COMMENTS

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

4/02R

FORM NO. TINUS 001

STANDARD OPERATING PROCEDURE
NUMBER CTO166-09

INSPECTION OF EXISTING MONITORING WELLS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes procedures for the inspection and repair of existing monitoring wells at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following equipment and field forms are required for inspection of existing monitoring wells.

Monitoring well inspection form: A copy of the monitoring well inspection form is attached.
Writing utensil with indelible ink
Bound field logbook
Well keys
Disposable medical-grade gloves (e.g., latex, nitrile)
Photoionization detector (PID)
Electronic water-level indicator
Steel rod (about 1 inch in diameter with eye bolt at one end)
100 feet of nylon rope
Internal pipe cutter
File, v notch

3.0 INSPECTION PROCEDURES

3.1 Record the well identification information (ID), date, and time on the Monitoring Well Inspection Form.

3.2 Record the condition of the well ID tag. Is the tag in place and legible? If not, note the discrepancies on the Monitoring Well Inspection Form.

3.3 Record the condition of the protective casing, caps, and lock. Has the casing, cap, and/or lock been tampered with or damaged? Has the well been damaged in any way or does it show signs of deterioration?

3.4 Record the condition of the concrete or gravel pads, if a pad is present. Check the condition of the pad (or the area around the well if no pad exists) and note any abnormalities. Are concrete pads cracking or heaving? If a gravel pad is present, is there any erosion or plant growth in the pad area?

3.5 Record the condition of the cement seal surrounding the protective casing. Has the seal cracked or pulled away from the protective casing? Record any visible signs of deterioration in the area of the seal.

3.6 Record the presence of depressions and/or standing water around the casing or pad.

3.7 Unlock the well cap and open the protective cover, if one exists.

3.8 Inspect and record the condition of the polyvinyl chloride (PVC) riser pipe and the surveyed reference point. The surveyed reference point is a V-notch on the top of the PVC riser pipe.

3.9 Measure the height of the protective casing and riser pipe above the ground surface. Record these readings on the inspection form to the nearest 0.01foot.

3.10 Check the existing well log to verify the total original depth of the monitoring well being inspected.

3.11 Open the well cap and use the PID to screen the air within the well opening to determine whether above-background levels of VOCs are present within the well. Refer to the Health & Safety Plan (HASP) for procedures to follow for the presence of VOCS in a well.

3.12 Lower the electronic water-level indicator probe down the well casing. If an obstruction is encountered, record the depth of the obstruction and whether the obstruction is partial or complete.

3.13 If no obstruction is encountered, continue lowering the water-level indicator down the well casing until ground water is encountered. Measure the depth to water to the nearest 0.01 foot (see SOP CTO166-18) and record the depth on the Inspection Log.

3.14 Continue lowering the indicator probe down the casing until a solid bottom is reached or an obstruction is encountered. Record the depth to the bottom of the well (from top of casing) on the Inspection Log.

3.15 If an obstruction is encountered in the casing before the well bottom is reached, record the depth of obstruction on the Inspection Log and whether the obstruction is partial or complete.

3.16 Remove the water-level indicator from the well.

3.17 If an obstruction was encountered during steps 3.12 or 3.14, lower a heavy steel rod slowly down the well casing until the obstruction is encountered. Attempt to loosen the obstruction by raising and dropping the steel rod, letting it hit the obstruction with gradually increasing force.. Record whether the obstruction could be loosened.

3.18 If step 3.17 is performed, remove the steel rod from the well and remeasure the depth to the obstruction. If the obstruction has been knocked loose and settles to the bottom, then the well may still be used as a piezometer but will not be used for ground water sampling.

3.19 Close the well cap and lock, if lock is present.

3.20 Decontaminate the water-level indicator and steel rod, if used, per SOP CTO166-17.

3.21 Make recommendations on the Inspection Log, if necessary, for repair of the monitoring well. Replace lock, if needed, as soon as possible.

3.22 Perform repair of well as soon as possible.

4.0 Field Repairs to Monitoring Wells

Field repairs to monitoring wells, for the most part, will be limited to the repair of the PVC riser (inter case). If a monitoring well is damaged beyond the repair capabilities of the field crew, the NSWC Crane Environmental Department will be notified and a decision will be made to repair or replace the well using a drilling contractor.

4.1 Remove a short section of the top of the PVC riser that is broken, using an inside pipe cutter. Before the riser is cut, a clean rag will be inserted into the riser to just below the cut to prevent

cuttings from entering the well. Insert the cutter just below the damaged area and remove only the damaged section of riser. Carefully remove the rag, measure and record the length of riser removed and mark the top of the riser for water-level measurement (filing a V notch is the preferred method).

4.2 If the riser damage indicates that a riser must be added, a section of PVC may be attached to the existing riser. After removing the damaged riser (see 4.1 above), cut a new section of riser to the needed length and attach a slip coupler to one end. In most cases, the coupler will fit securely to the pipe without the need for mechanical fasteners (pop-rivets/screws). If needed, fasteners may be used, but in no case will glue be used to attach the coupler. Attach the new coupler and PVC pipe to the existing riser, mark the top for measurement purposes, and document the length of pipe added. All new materials (pipe and couplers) will be decontaminated prior to installation.

5.0 ATTACHMENTS

1. Monitoring Well Inspection Sheet

ATTACHMENT 1

MONITORING WELL INSPECTION SHEET

ATTACHMENT 1

MONITORING WELL INSPECTION SHEET

MONITORING WELL INSPECTION SHEET

Well ID: _____

Time: _____ **Date:** _____ **Inspector's Name:** _____

Inspection Item	Types of Problems	Status		Observation
		S	U	
Well Tag	Is it in-place, legible			
Well security	Condition protective case, cap, lock			
Well pad	Concrete or gravel & condition			
Well seal	Condition of...			
Area Immediately around well pad	Record any evidence of/or standing water in area of well			
Dedicated sampling equipment	Condition of...			
PVC Riser	Condition of riser & survey reference point			

Comments:

Signature(s)_____

Note: S= Satisfactory, U= Unsatisfactory
Check one, if unsatisfactory explain

STANDARD OPERATING PROCEDURE
NUMBER CTO166-10

MONITORING WELL DEVELOPMENT

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper development of new and existing monitoring wells. The methods described herein are specific for monitoring wells located at the NSWC Crane facility. Guidelines by South Division, Naval Facilities Engineering Command, (SOUTHDIV NAVFAC, 1997) and the State of Indiana regulatory requirements in Article 16 Water Well Drillers of Chapter 310 of the Indiana Annotated Codes (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

The drilling contractor will provide adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of developing monitoring wells. The drilling contractor personnel must have all the health and safety training required to perform the work, as specified in the health and safety plan (HASP).

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the site safety officer.

Well development equipment with associated materials (typically supplied by the driller).

Hydrogeologic equipment (weighted engineer's tape, water-level indicator, retractable engineers rule, electronic calculator, clipboard, mirror and flashlight for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, and a field notebook).

4.0 WELL DEVELOPMENT METHODS

The development of new wells will not occur until at least 48 hours after the well has been installed and grouted. This time is required so that the grout in the annulus can set and harden. The purpose of well development is to stabilize and increase the permeability of the sand pack and the well screen and to restore the permeability of the formation that may have been reduced by drilling operations. Wells are typically developed until all fine material and drilling water, if any, is removed from the well.

Sequential measurements of pH, specific conductance, turbidity, and temperature taken during development yield information (stabilized values) that sufficient development is reached. Development should proceed until criteria are met as stated in Navy Guidelines.

A surge plunger (also called a surge block) that is approximately the same diameter as the well casing will be used to agitate the water, causing it to move in and out of the screens. This movement of water pulls fine materials into the well, where they may be removed by any of several methods, and prevents bridging of sand particles in the gravel pack. There are two basic types of surge plungers, solid and valved surge plungers. Site-specific conditions will dictate which type will be used. In formations with low yields, a valved surge plunger may be preferred because solid plungers tend to force water out of the well at a greater rate than it will flow back in. Valved plungers are designed to produce a greater inflow than outflow of water during surging.

Development should proceed until the following criteria are met:

- The well water is clear to the unaided eye.

and

- A minimum removal of five times the standing water volume in the well (to include the well screen and casing plus saturated borehole annulus, assuming 30% annular porosity).

or

- When pH measurements remain constant within 0.1 Standard Units and specific conductance and temperature vary no more than plus or minus 3 percent for at least three consecutive readings. Turbidity should also show stabilization and ideally be below 10 nephelometric turbidity units (NTUs).

If for any reason the above criteria cannot be met, the site geologist should document the event in writing and consult with the Task Order Manager regarding an alternate plan of action.

Well development must be completed at least 24 hours before well sampling. The intent of this hiatus is to provide time for the newly installed well and backfill materials to sufficiently equilibrate to their new environment and for the new environment to re-stabilize after the disturbance of drilling.

5.0 ATTACHMENTS

1. Monitoring Well Development Record

ATTACHMENT 1

MONITORING WELL DEVELOPMENT RECORD

Tetra Tech NUS, Inc.

MONITORING WELL DEVELOPMENT RECORD

PROJECT: _____ WELL No.: _____

PROJECT No.: _____ DATE INSTALLED: _____

SITE: _____ DATE DEVELOPED: _____

PROJECT GEOLOGIST: _____ DRILLING Co.: _____

WELL DATA

WELL MATERIAL: ☐ PVC ☐ STAINLESS STEEL ☐ OTHER: _____

TOTAL DEPTH: _____ feet (TOC / bgs) CASING ID: _____ inches

STATIC WATER LEVEL: _____ feet (TOC / bgs) SCREEN LENGTH: _____ feet

CASING VOLUME _____ (gal. / L) BOREHOLE DIA.: _____ inches

PURGE DATA

START TIME: _____ (hr.) METHOD: _____

STOP TIME: _____ (hr.) PUMP TYPE: _____

Time	Cumulative Volume (gal. / L)	Pumping Rate (gpm / Lpm	Water Level (ft. / m.)	Water Meter Type: pH	Sp. Cond. (mS/cm)	Temp. (°C)	Turbidity (NTU)	DO (mg/L)	Remarks (Color/Odor)

TOTAL PURGE VOLUME: (gal. / L) TOTAL PURGE TIME: (min. / hrs.

NOTE: All depth measurements to nearest 0.01 foot.

COMMENTS/CALCULATIONS:

STANDARD OPERATING PROCEDURE
NUMBER CTO166-11

DRILLING AND GEOLOGIC LOGGING OF BOREHOLES IN ROCK

1.0 PURPOSE

This procedure describes the methods and equipment necessary to drill rock borings and identify the equipment, sequence of events, and appropriate methods necessary to obtain rock cores and prepare boring logs during drilling activities. Up to four types of drilling activities and equipment will be used to drill holes and install monitoring wells at the NSWC Crane facility:

- Auger drilling will be used to drill through the overburden material.

- Diamond coring equipment will be used to core through the bedrock. NX or similar size diamond core barrels will be used to collect 2- to 3-inch-diameter continuous rock core. These cores will be used to describe the lithologic characteristics and fracture distributions in the bedrock. Diamond coring is relatively slow and more expensive compared to the air rotary method of drilling, so only two holes are being cored.

- Air-rotary drilling will be used to ream out to a larger diameter in the two diamond-core holes mentioned above and to drill the other two deep well borings described in the QAPP. The holes need to be a minimum of 5 inches in diameter in order to install 2-inch-diameter monitoring wells. Air rotary may also be used to deepen well borings in the event bedrock is encountered before the desired well boring depth is reached.

- Rotary drilling with a roller bit and water wash may be used to complete well borings in the event that bedrock is encountered before the desired well boring depth is reached.

2.0 RESPONSIBILITIES

Field Operations Leader (FOL) - The FOL is responsible for coordinating all on-site personnel and for providing technical assistance, when required. The FOL, or designee, will coordinate and lead all activities and will ensure the availability and maintenance of all materials and equipment. The FOL is responsible for the completion of all field activities and field and chain-of-custody documentation. The

FOL will assume custody of all samples and will ensure the proper handling and shipping of samples. The FOL is a highly experienced environmental professional who will report directly to the TtNUS Task Order Manager (TOM). Specific FOL responsibilities include the following:

- Function as a communications link among field staff members, the site quality assurance/quality control advisor, site safety officer, the site manager, and the TOM.
- Oversee the mobilization and demobilization of all field equipment and subcontractors.
- Coordinate and manage the field technical staff.
- Adhere to the work schedules provided by the TOM.
- Maintain the site logbook and field recordkeeping.
- Initiate field task modification requests, when necessary.
- Identify and resolve problems in the field, resolve difficulties in consultation with the NSWC Crane Site Manager, implement and document corrective action procedures, and provide communication between the field team and upper management.

Field Geologist - The field geologist is responsible for ensuring that standard and approved drilling procedures are followed. The field geologist will generate a detailed boring log for each borehole. This log will include a description of geologic materials, samples (if any), method of sampling, and other pertinent information and observations that may be obtained during drilling.

Determination of the exact location for borings is the responsibility of the field geologist. The final location for drilling must be properly documented on the boring log. The general area in which the borings are to be located will be shown on a site map included in the QAPP.

Drilling Subcontractor - The subcontractor operates under the supervision of the FOL. He or she is responsible for obtaining all drilling permits and clearances and supplying all services (including labor), equipment, and material required to perform the drilling, testing, and well installation program, as well as maintenance and quality control of such required equipment except as stated in signed and approved subcontracts.

The driller must report any major technical problems encountered in the field to the FOL within 24 hours of determination and must provide advance written notification of any changes in field procedures, describing and justifying such changes. No such changes will be made unless requested and authorized in writing by the FOL (with the concurrence of the project manager). Depending on the subcontract, the project manager may need to obtain written authorization from appropriate administrative personnel before approving any changes.

The drilling subcontractor is responsible for following decontamination procedures specified for drilling and coring equipment specified in the project plan documents. The FOL will oversee the in-field equipment decontamination procedures to confirm compliance with the appropriate SOP and specific requirements of the NSWC Crane Environmental Department. Upon completion of the work, the driller is responsible for demobilizing all equipment, cleaning up any materials deposited on site during drilling operations, and properly backfilling any open borings.

3.0 PROCEDURES

3.1 General

The purpose of drilling boreholes is

- To determine the type, thickness, and certain physical and chemical properties of the soil, water, and rock strata that underlie the site.
- To install monitoring wells or piezometers.

All drilling and sampling equipment will be cleaned between samples and borings using appropriate decontamination procedures as outlined in SOP CTO166-17. Unless otherwise specified, it is generally advisable to drill borings at "clean" locations first and at the most contaminated locations last to reduce the risk of spreading contamination between locations. All borings must be logged by the site geologist as they proceed.

3.2 Rock Coring

Drilling is done by rotating and applying downward pressure to the drill rods and drill bit. The drill bit is a circular, hollow, diamond-studded bit attached to the outer core barrel in a double-tube core barrel. The use of single-tube core barrels is not recommended because the rotation of the barrel erodes the sample and limits its use for detailed geological evaluation. Water or air is circulated down through the drill rods

and annular space between the core barrel tubes to cool the bit and remove the cuttings. The bit cuts a core out of the rock that rises into an inner barrel mounted inside the outer barrel. The inner core barrel and rock core are removed by lowering a wire line with a coupling into the drill rods, latching onto the inner barrel, and withdrawing the inner barrel. A less efficient variation of this method utilizes a core barrel that cannot be removed without pulling all the drill rods. This variation is practical only if fewer than 50 feet of core is required. When coring rock, the speed of the drill and the drilling pressure, amount and pressure of water, and length of run can be varied to give the maximum recovery from the rock being drilled.

Advantages of core drilling include

- Undisturbed rock cores can be recovered for examination and/or testing.
- In formations in which the cored hole will remain open without casing, water from the rock fractures may be recovered from the well without the installation of a well screen and gravel pack.
- Formation logging is extremely accurate.
- Drill rigs are relatively small and mobile.

Disadvantages include

- Water or air is needed for drilling.
- Coring is slower than rotary drilling (and more expensive).
- Depth to water cannot accurately be determined if water is used for drilling.
- The size of the borehole is limited.

This drilling method is useful if accurate determinations of rock lithology are desired or if open wells are to be installed into bedrock. To install monitoring wells in coreholes, the hole will be reamed out to the proper size after boring, using air rotary drilling methods. Rock coring enables a detailed assessment of borehole conditions to be made, showing precisely all lithologic changes and characteristics. Because coring is an expensive drilling method, it is commonly used for shallow studies of 500 feet or less or for specific intervals in the drill hole that require detailed logging and/or analyzing. Rock coring can, however, proceed for thousands of feet continuously, depending on the size of the drill rig, and can yield better quality data than air-rotary drilling, although at a substantially reduced drilling rate.

Borehole diameter can be drilled to various sizes, depending on the information needed. NX, or a similar size (2- to 3-inch-diameter core recovery), may be used.

Begin the core drilling using a double-tube swivel-core barrel of the desired size. After drilling no more than 10 feet (3 meters), remove the core barrel from the hole and take out the core. If the core blocks the flow of the drilling fluid during drilling, remove the core barrel immediately.

Since rock structures and the occurrence of bedding planes, porosity type and distribution, and fracture patterns are among the most important items to be detected and described, take special care to obtain and record these features. If such broken zones or cavities prevent further advance of the boring, one of the following three steps shall be taken: cement the hole , ream and case, or case and advance with the next smaller size core barrel, as conditions warrant.

3.3 Rock Core Management and Labeling

When the core barrel has been recovered, the rock core will be carefully removed from the barrel, placed in a core tray (previously labeled "top" and "bottom" to avoid confusion), classified, and measured for percentage of recovery as well as the rock quality designation (RQD). Each core will be described, classified, and logged using a uniform system (Section 3.5 of this SOP).

Rock cores will be placed in the sequence of recovery in well-constructed wooden or cardboard boxes provided by the drilling contractor. Rock cores from two different borings will not be placed in the same core box. The core boxes will be constructed to accommodate at least 20 linear feet of core in rows of approximately 5 feet each. Wood partitions will be placed at the end of each core run. The depth from the surface of the boring to the top and bottom of the drill run and run number will be marked on the wooden partitions with indelible ink. These blocks will serve to separate successive core runs and indicate depth intervals for each run. The order of placing cores will be the same in all core boxes. Rock core will be placed in the box so that, when the box is open, with the inside of the lid facing the observer, the top of the cored interval contained within the box is in the upper left corner of the box and the bottom of the cored interval is in the lower right corner of the box. The top and bottom of each core obtained and their true depths will be clearly and permanently marked on each box. The width of each row must be compatible with the core diameter to prevent lateral movement of the core in the box. Similarly, an empty space in a row will be filled with an appropriate filler material or spacers to prevent longitudinal movement of the core in the box.

The inside and outside of the core-box lid will be marked by indelible ink to show all pertinent data about the box's contents. At a minimum, the following information will be included:

- Project name
- Project number
- Boring number
- Run numbers
- Footage (depths)
- Recovery
- RQD (%)
- Box number and total number of boxes for that boring (Example: Box 5 of 7)
- Contact person's name and telephone number

For easy retrieval when core boxes are stacked, the sides and ends of the box will also be labeled and will include project number, boring number, top and bottom depths of core, and box number.

Before final closing of the core box, a photograph will be taken of the recovered core and the labeling on the inside cover will be taken. If moisture content is not critical, the core shall be wetted and wiped clean for the photograph. (This will help to show true colors and bedding features in the cores.)

3.4 Air Rotary Drilling

Air-rotary drilling is a method of drilling where the drill rig simultaneously turns and exerts a downward pressure on the drilling rods and bit while circulating compressed air down the inside of the drill rods, around the bit, and out the annulus of the borehole. Air circulation serves to both cool the bit and remove the cuttings from the borehole.

Advantages of this method include

- The drilling rate is high (even in rock).
- The cost per foot of drilling is relatively low.
- Air-rotary rigs are common in most areas.
- No drilling fluid is required (except when water is injected to keep down dust).
- The borehole diameter is large to allow room for proper well installation procedures.

Disadvantages to using this method include

- Formations must be logged from the cuttings that are blown to the surface and thus the depths of materials logged are approximate.

- Air blown into the formation during drilling may "bind" the formation and impede well development and natural groundwater flow.

- In-situ samples cannot be taken, unless the hole is cased.

- Air-rotary drill rigs are large and heavy.

- Large amounts of investigation-derived waste (IDW) may be generated that may require containerization, sampling, and off-site disposal.

4.0 GEOLOGIC DESCRIPTION OF BOREHOLES IN ROCK

These procedures provide descriptions of the standard techniques for borehole and sample logging. These logging techniques will be used for each boring to provide consistent descriptions of subsurface lithology. While experience is the only method to develop confidence and accuracy in the description of soil and rock, the field geologist or engineer can do a good job of classification by careful, thoughtful observation and by being consistent throughout the classification procedure.

The classification of soil and rocks is one of the most important jobs of the field geologist or engineer. To maintain a consistent flow of information, it is imperative that the field geologist or engineer understand and accurately use the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 Required Field Forms and Equipment

When logging soil and rock samples, the geologist or engineer should be equipped with the following:

Rock hammer
Knife
Camera
10% Dilute hydrochloric acid (HCl)
Ruler (marked in tenths and hundredths of feet)

Hand lens

Writing utensil with indelible ink

Field logbook

Disposable medical-grade gloves (e.g., latex, nitrile)

Soil/rock classification sheets

4.2 Classification of Rocks

Claystone - Very fine-grained rock made up of particle less than 1/256 mm in diameter. Fractures irregularly. Very smooth to touch. Generally has irregularly spaced pitting on surface of drilled cores.

Shale - A fissile, very fine-grained rock with particles less than 1/256 mm in diameter. Fractures along bedding planes.

Limestone - Rock made up predominantly of calcite ($CaCO_3$, which is mainly fossilized animal and plant debris). Effervesces strongly upon the application of dilute hydrochloric acid.

Coal – A very dark colored rock consisting mainly of organic (mainly fossilized plant debris) remains.

Others - Numerous other sedimentary rock types are present in lesser amounts in the stratigraphic record.

The local abundance of any of these rock types is dependent upon the depositional history of the area. Conglomerate, halite, gypsum, dolomite, anhydrite, lignite, etc. are some of the rock types found in lesser amounts.

In classifying a sedimentary rock, the following hierarchy will be noted: Rocks are grouped into three main divisions: sedimentary, igneous, and metamorphic. Sedimentary rocks are by far the predominant type exposed at the earth's surface and are the only type present at NSWC Crane. The following basic names are applied to the types of rocks found in sedimentary sequences:

Sandstone - Made up predominantly of granular materials ranging between 1/16 to 2 mm in diameter.

Siltstone - Made up of granular materials between 1/16 to 1/256 mm in diameter. Fractures irregularly. Medium thick to thick bedded.

4.2.1 Rock Type

As described above, there are numerous types of sedimentary rocks. In most cases, a rock will be a combination of several grain types; therefore, a modifier such as "sandy siltstone" or a "silty sandstone" can be used. The modifier indicates that a significant portion of the rock type is composed of the modifier. Other modifying descriptions can include carbonaceous, calcareous, siliceous, fossiliferous, etc.

Grain size is the basis for the classification of clastic (sandstones, siltstones, and shales) sedimentary rocks. The Udden-Wentworth classification will be assigned to sedimentary rocks (shown below). The individual boundaries are slightly different than the USGS subdivision for soil classification. For field determination of grain sizes, a scale can be used for the coarse-grained rocks. Alternatively, the division between siltstone and shale may be measurable in the field by the use of a hand lens. If the grains cannot be seen with the naked eye but are distinguishable with a hand lens, the rock is a siltstone. If the grains are not distinguishable with a hand lens, the rock is a shale.

GRAIN SIZE CLASSIFICATION FOR ROCKS

Particle Name	Grain Size Diameter
Cobbles	> 64 mm
Pebbles	4 - 64 mm
Granules	2 - 4 mm
Very Coarse Sand	1 - 2 mm
Coarse Sand	0.5 - 1 mm
Medium Sand	0.25 - 0.5 mm
Fine Sand	0.125 - 0.25 mm
Very Fine Sand	0.0625 - 0.125 mm
Silt	0.0039 - 0.0625 mm
After Wentworth, 1922	

4.2.2 Color

The color of a rock can be determined in a similar manner as for soil samples. Rock core samples will be classified while wet, when possible, and air-cored samples will be scraped clean of cuttings prior to color classifications. Soil colors will be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Since color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

4.2.3 Bedding Thickness

The bedding thickness designations listed below will also be used for rock classification.

BEDDING THICKNESS CLASSIFICATION

Thickness (metric)	Thickness (Approximate English Equivalent)	Classification
> 1.0 meter	> 3.3'	Massive
30 cm - 1 meter	1.0' - 3.3'	Thick bedded
10 cm - 30 cm	4" - 1.0'	Medium bedded
3 cm - 10 cm	1" - 4"	Thin bedded
1 cm - 3 cm	2/5" - 1"	Very thin bedded
3 mm - 1 cm	1/8" - 2/5"	Laminated
1 mm - 3 mm	1/32" - 1/8"	Thinly laminated
< 1 mm	<1/32"	Micro laminated

(Ingram, 1954)

4.2.4 Hardness

The hardness of a rock is a function of the compaction, cementation, and mineralogical composition of the rock. A relative scale for sedimentary rock hardness is as follows:

Soft - Weathered, considerable erosion of core, easily gouged by screwdriver, scratched by fingernail. Soft rock crushes or deforms under pressure of a pressed hammer. This term is always used for the hardness of the saprolite (decomposed rock that occupies the zone between the lowest soil horizon and firm bedrock).

Medium soft - Slight erosion of core, slightly gouged by screwdriver, or breaks with crumbly edges from single hammer blow.

Medium hard - No core erosion, easily scratched by screwdriver, or breaks with sharp edges from single hammer blow.

Hard - Requires several hammer blows to break and has sharp conchoidal breaks. Cannot be scratched with screwdriver.

Note the difference in usage of the words "scratch" and "gouge." A scratch will be considered a slight depression in the rock (do not mistake the scraping off of rock flour from drilling with a scratch in the rock itself), and a gouge is much deeper.

4.2.5 Fracturing

Method of Calculating RQD
(After Deere, 1966)

Fractures should also be noted.

$$RQD\ \% = r/l \times 100$$

r = Total length of all pieces of the lithologic unit being measured that are greater than 4 inches in length and have resulted from natural breaks. Natural breaks include slickensides, joints, compaction slicks, bedding plane partings (not caused by drilling), friable zones, etc.

l = Total length of the coring run.

4.2.6 Weathering

The degree of weathering is a significant parameter that is important in determining weathering profiles and is also useful in engineering designs. The following terms can be applied to distinguish the degree of weathering:

Fresh - The rock shows little or no weathering effect. Fractures or joints have little or no staining and rock has a bright appearance.

Slight - The rock has some staining that may penetrate several centimeters into the rock. Clay filling of joints may occur. Feldspar grains may show some alteration. Oxidation and weathering may affect the degree of fracturing or brokenness of a rock. After eliminating drilling breaks, the average spacing of natural breaks is calculated and the fracturing is described by the following terms:

Very broken (V. BR.) - Less than 2-inch spacing between fractures
Broken (BR.) - 2-inch to 1-foot spacing between fractures

Blocky (BL.) - 1- to 3-foot spacing between fractures
Massive (M.) - 3- to 10-foot spacing between fractures

The structural integrity of the rock can be approximated by calculating the rock quality designation (RQD) of cores recovered. The RQD is determined by adding the total lengths of all pieces exceeding 4 inches and dividing by the total length of the coring run to obtain a percentage.

Moderate - Most of the rock, with exception of quartz grains, is stained. Rock is weakened due to weathering and can be easily broken with a hammer.

Severe - All rock including quartz grains is stained. Some of the rock is weathered to the extent of becoming a soil. Rock is very weak.

4.2.7 Other Characteristics

The following items will be included in the rock description:

Description of contact between two rock units. These can be sharp or gradational.

- Stratification (parallel, cross stratified).
- Description of any filled cavities or vugs.
- Cementation (calcareous, siliceous, hematitic).
- Description of any joints or open fractures.
- Observation of the presence of fossils.
- Notation of joints with depth, approximate angle to horizontal, any mineral filling or coating, and degree of weathering.

All information shown on the boring logs will be neat so it can be reproduced on a copy machine for report presentation. The data will be kept current to provide control of the drilling program and to indicate various areas requiring special consideration and sampling.

4.2.8 Additional Terms Used in the Description of Rock

The following terms are used to further identify rocks:

Seam - Thin (12 inches or less), probably continuous layer.

Some - Indicates significant (15 to 40 percent) amounts of the accessory material. For example, rock composed of seams of sandstone (70 percent) and shale (30 percent) would be "sandstone -- some shale seams."

Few - Indicates insignificant (0 to 15 percent) amounts of the accessory material. For example, rock composed of seam of sandstone (90 percent) and shale (10 percent) would be "sandstone -- few shale seams."

Interbedded - Used to indicate thin or very thin alternating seams of material occurring in approximately equal amounts. For example, rock composed of thin alternating seams of sandstone (50 percent) and shale (50 percent) would be "interbedded sandstone and shale."

Interlayered - Used to indicate thick alternating seams of material occurring in approximately equal amounts.

4.2.9 Abbreviations

Abbreviations may be used in the description of a rock. However, they will be kept at a minimum. Following are some of the abbreviations that may be used:

C - Coarse	Lt - Light	Yl - Yellow
Med - Medium	BR - Broken	Or - Orange
F - Fine	BL - Blocky	SS - Sandstone
V - Very	M - Massive	Sh - Shale
Sl - Slight	Br - Brown	LS - Limestone
Occ - Occasional	Bl - Black	Fgr - Fine-grained
Tr - Trace		

5.0 BORING LOGS AND DOCUMENTATION

This section describes in more detail the procedures to be used in completing boring logs in the field. Information obtained from the preceding sections will be used to complete the logs. A sample boring log is attached at the end of this SOP.

The field geologist or engineer will use this example as a guide in completing each boring log. Each boring log will be fully described by the geologist or engineer as the boring is being drilled. Every sheet

contains space for 25 feet of log. Information regarding classification details is provided either on the back of the boring log or on a separate sheet, for field use. All data will be written directly on the boring log. Additional notes may be entered in a field notebook if more space is needed.

5.1 Remarks Column

The following information will be entered under the "Remarks" column and will include, but is not limited to, the following:

- Moisture - Estimate moisture content using the following terms: dry, moist, wet, and saturated. These terms are determined by the individual. Whatever method is used to determine moisture should be consistent throughout the log.

- Angularity - Describe angularity of coarse-grained particles using the terms angular, subangular, subrounded, or rounded. Refer to ASTM D 2488 or the Earth Manual for criteria for these terms.

- Particle shape - flat, elongated, or flat and elongated.

- Maximum particle size or dimension.

- Water-level observations.

- Reaction with HCl - none, weak, or strong.

Additional comments:

- Indicate presence of mica, caving of hole, when water was encountered, difficulty in drilling, loss or gain of water.

- Indicate odor and photoionization detector (PID) readings.

- Indicate any change in lithology by drawing a line through the lithology change column and indicate the depth. This will help when cross-sections are subsequently constructed.

- At the bottom of the page, indicate type of rig, drilling method, hammer size and drop, and any other useful information (i.e., borehole size, casing set, changes in drilling method).

- Vertical lines shall be drawn in the Material Description column from the bottom of each sample to the top of the next sample to indicate consistency of material from sample to sample, if the material is consistent. Horizontal lines will be drawn if there is a change in lithology, then vertical lines will be drawn to that point.

- Indicate screened interval of well, as needed, in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.2 Rock Classification

Indicate depth at which coring began by drawing a line at the appropriate depth. Indicate core run depths by drawing coring run lines (as shown) under the first and fourth columns on the log sheet. Indicate core run number, RQD percent, and core recovery under the appropriate columns.

Indicate lithology change by drawing a line at the appropriate depth, as explained above.

Rock hardness is entered under designated column using terms as described on the back of the log or as explained earlier in this section.

Enter color as determined while the core sample is wet; if the sample is cored by air, the core is to be scraped clean before the color is described.

Enter rock type based on sedimentary, igneous, or metamorphic. For sedimentary rocks, use terms as described in Section 5.3. Again, be consistent in classification. Use modifiers and additional terms as needed. For igneous and metamorphic rock types, use terms as described in Section 5.3.

Enter brokenness of rock or degree of fracturing under the appropriate column using symbols VBR, BR, BL, or M, as explained in Section 5.3 and as noted on the back of the boring log.

The following information will be entered under the remarks column. Items will include but are not limited to the following:

- Indicate depths of joints, fractures, and breaks and also approximate to horizontal angle (such as high, low) (i.e., 70° angle from horizontal, high angle).
- Indicate calcareous zones, description of any cavities or vugs.
- Indicate any loss or gain of drill water.
- Indicate drop of drill tools or change in color of drill water.

Remarks at the bottom of boring log will include

- Type and size of core obtained
- Depth casing was set
- Type of rig used

As a final check, the boring log will include the following:

- Vertical lines will be drawn as explained for soil classification to indicate consistency of bedrock material.

- If applicable, indicate screened interval in the lithology column. Show top and bottom of screen. Other details of well construction are provided on the well construction forms.

5.3 Classification of Soil and Rock from Drill Cuttings

The previous sections describe procedures for classifying rock samples when cores are obtained. However, some drilling methods (air/mud rotary) may require classification and borehole logging based on identifying drill cuttings removed from the borehole. Such cuttings provide only general information on subsurface lithology. Some procedures that will be followed when logging cuttings are as follows:

- Obtain cutting samples at approximately 5-foot intervals, sieve the cuttings (if mud rotary drilling) to obtain a cleaner sample, place the sample into a small sample bottle or ziplock bag for future reference, and label the jar or bag (i.e., hole number, depth, date, etc.). Cuttings will be closely examined to determine general lithology.

- Note any change in color of drilling fluid or cuttings to estimate changes in lithology.

- Note drop or chattering of drilling tools or a change in the rate of drilling to determine fracture locations or lithologic changes.

- Observe loss or gain of drilling fluids or air (if air rotary methods are used) to identify potential fracture zones.

- Record this and any other useful information onto the boring log.

This logging provides a general description of subsurface lithology, and adequate information can be obtained through careful observation of the drilling process. It is recommended that split-barrel and rock core sampling methods be used at selected boring locations during the field investigation to provide detailed information to supplement the less-detailed data generated through borings drilled using air and mud rotary methods.

5.4 Review

Upon completion of the borings logs, copies will be made and reviewed. Items to be reviewed include

- Checking for consistency of all logs
- Checking for conformance to the guideline
- Checking to see that all information is entered in their respective columns and spaces

Originals of the boring logs will be retained in the project files.

7.0 ATTACHMENTS

1. Boring Log

ATTACHMENT 1

BORING LOG

Tetra Tech NUS, Inc.

BORING LOG

Page ___ of ___

PROJECT NAME: _____ BORING No.: _____
PROJECT NUMBER: _____ DATE: _____
DRILLING COMPANY: _____ GEOLOGIST: _____
DRILLING RIG: _____ DRILLER: _____

Sample No. and Type or RQD	Depth (Ft.) or Run No.	Blows / 6" or RQD (%)	Sample Recovery / Sample Length	Lithology Change (Depth/Ft.) or Screened Interval	MATERIAL DESCRIPTION: Soil Density/ Consistency or Rock Hardness	MATERIAL DESCRIPTION: Color	MATERIAL DESCRIPTION: Material Classification	USCS *	Remarks	PID/FID Reading (ppm): Sample	PID/FID Reading (ppm): Sampler BZ	PID/FID Reading (ppm): Borehole**	PID/FID Reading (ppm): Driller BZ**

* When rock coring, enter rock brokeness.

** Include monitor reading in 6 foot intervals @ borehole. Increase reading frequency if elevated reponse read.

Drilling Area Background (ppm): [ ]

Remarks: _____

Converted to Well: Yes No Well I.D. #:

References

Deere, D.U. and Miller, R.P., (1966). Engineering Classification and Index Properties for Intact Rock, Air Force Weapons Lab. Tech. Report AFWL-TR-65-116, Kirtland Base, New Mexico.

Wentworth, C.K., 1922. A Scale of Grade and Class Terms for Caustic Sediments: Jour. Geology, v. 30, p. 377-392.

Ingram, R.L., 1954. Terminology for the Thickness of Stratification and Parting Units in Sedimentary Rocks: Geological Society of America Bulletin, v. 65. p. 937-938.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-12

MONITORING WELL INSTALLATION

1.0 PURPOSE

This procedure provides general guidance and information pertaining to proper design and installation of ground water monitoring wells. The methods described herein are specific for monitoring well construction at the NSWC Crane facility. Guidelines by South Division, Naval Facilities Engineering Command (SOUTHNAVFACENGCOM, 1997) and the State of Indiana regulatory requirements in Article 16 Water Well Drillers of Chapter 310 of the Indiana Annotated Codes (310 IAC 16) should be consulted.

2.0 RESPONSIBILITIES

Driller - The driller provides adequate and operable equipment, sufficient quantities of materials, and an experienced and efficient labor force capable of performing all phases of proper monitoring well installation and construction. The drilling contractor personnel must have all the health and safety training required to perform the work, as specified in the health and safety plan. The driller is also responsible for obtaining, in advance, any required permits for drilling and monitoring well installation and construction.

Field Geologist - The field geologist supervises and documents well installation and construction performed by the driller and ensures that the screen interval for each monitoring well is properly placed to provide representative groundwater data from the monitored interval. Geotechnical engineers, field technicians, or other suitable trained personnel may also serve in this capacity.

Site Safety Officer – The site safety officer is responsible for clearing the drill site for underground and overhead utilities or other potentially hazardous obstructions.

3.0 REQUIRED EQUIPMENT/ITEMS

The following list includes equipment and items required for monitoring well installation:

Health and safety equipment as required by the HASP and the site safety officer.

Well drilling and installation equipment with associated materials (typically supplied by the driller).

Hydrogeologic equipment (weighted engineer's tape, water-level indicator, retractable engineer's rule, electronic calculator, clipboard, mirror and flashlight for observing downhole activities, paint and ink marker for marking monitoring wells, sample jars, well installation forms, boring logs, soil sample log forms, chain-of-custody records, sample coolers with ice, and a field notebook).

4.0 WELL DESIGN AND CONSTRUCTION

Overburden well borings will be drilled using minimum 4-inch inside diameter hollow-stem augers to the desired depth of each well boring. Rotary drilling with a roller bit and water wash or air rotary drilling can be used to extend the shallow well borings, in the event that bedrock comprising aquifer material is encountered before the desired well depth is reached. In no cases will the Elwren shale be completely penetrated unless a steel casing is installed and grouted in place at the top of the Elwren shale.

Bedrock monitoring well borings will be drilled using hollow-stem auger or rotary drilling techniques until the bedrock is reached. A 6-inch-diameter steel casing will be installed to the top of the bedrock and will be grouted with a cement-bentonite slurry in a manner to ensure that the entire annulus between the casing and borehole is sealed. Diamond coring (NX) will be initiated after the grout is allowed to cure for a minimum of 24 hours in those borings where coring is planned. For each well, the coring will proceed from the bottom of the casing to the full depth of the boring. Once the coring has been completed and the core has been logged (see SOP CTO166-11), then the hole will be reamed out with a minimum 5-inch-diameter air rotary bit. Air rotary drilling will be performed through the casing immediately after the grout has cured in those cases where coring is not anticipated. These borings will be logged using the rock chips and dust blown up the boring by the return air of the drill bit (see SOP CTO166-11). The borehole will then be cleaned out using compressed air from the drill bit. The air rotary equipment must have a filter on the compressed air line going to the borehole to prevent oil and other organics from being introduced. Once the hole has been cleaned out using the compressed air of the rig, packer testing of the hole can proceed (see SOP CTO166-13). Installation of the monitoring well follows the packer testing. Note: All drilling and packer testing equipment must be decontaminated before it is placed in a borehole.

All monitoring wells will be constructed of Schedule 40, flush-joint-threaded, 2-inch inside diameter polyvinyl chloride (PVC) riser pipe and flush joint threaded, factory slotted well screen with a threaded end cap. The well screens will be factory slotted to 0.020-inch size. Each section of well casing and screen will be National Sanitation Foundation (NSF) approved. Well screens will be 10 feet long but may be longer or shorter based on the subsurface conditions that are encountered. A PVC cap will be placed

on the bottom and will also be flush threaded. Other means of joining casings using glue, gaskets, pop rivets, or screws are not allowed. The screen will pass no more than 10 percent of the pack material or in-situ aquifer material.

Monitoring wells will be installed immediately upon completion of drilling or packer testing, if performed. A well screen section with bottom cap and the proper amount of riser pipe will be assembled and lowered down the borehole. Centralizers will be used as necessary to ensure that the casing and screen are centered and are aligned straight. The sand pack will be extended from 0.5 foot below the well screen to 2.0 feet above the top of the well screen. Clean silica sand of U.S. Standard Sieve Size No. 20 to 40 will be used.

A minimum 3-foot-thick bentonite pellet seal will be installed above the filter pack and allowed to hydrate for a minimum of 3 hours before grout is added above the seal. Only 100 percent, certified pure sodium bentonite will be used for well construction. The depths of backfill materials will be constantly monitored during well installation using a weighted stainless-steel or fiberglass tape measure.

The remaining annulus above the hydrated bentonite seal will be backfilled to the surface using a tremie pipe, with a 20:1 cement/bentonite grout. A maximum of 10 gallons of water per 94-pound bag of Type 1 cement will be used. The grout mixture should be blended in an above-ground rigid container or mixer to produce a thick lump-free mixture. This grout mixture will also be used for placement of steel casings in deep wells.

Bentonite expands by absorbing water and provides a seal between the screened interval and the overlying portion of the annular space and formation. Cement-bentonite grout is placed on top of the bentonite pellets extending to the surface. The grout effectively seals the well and eliminates the possibility for surface infiltration reaching the screened interval. Grouting also replaces material removed during drilling and prevents hole collapse and subsidence around the well. A tremie pipe should be used to introduce grout from the bottom of the hole upward to prevent bridging and to provide a better seal. However, in shallow boreholes that do not collapse, it may be more practical to pour the grout from the surface without a tremie pipe.

When the well is completed and grouted to the surface, a protective steel surface casing is placed over the top of the riser pipe. The finished well casing will extend at least 2 feet above the ground level. This casing will have a cap that can be locked to prevent vandalism. A vent hole will l be provided in the cap to allow venting of gases and maintain atmospheric pressure as water levels rise or fall in the well. The protective casing has a larger diameter than the riser pipe and is set into the wet cement grout over the

well upon completion or can be placed between the steel casing and the riser pipe in the case of deep monitoring well borings. In addition, one hole is drilled just above the cement collar through the protective casing, which acts as a weep hole for the flow of water that may enter the annulus during well development, purging, or sampling.

5.0 DOCUMENTATION OF FIELD ACTIVITIES

A critical part of monitoring well installation is recording of significant details and events in the site logbook, on field forms, and in a field logbook. Details of borehole logging are contained in SOP CTO166-11.

6.0 ATTACHMENTS

1. Bedrock Monitoring Well Sheet
2. Overburden Monitoring Well Sheet

ATTACHMENT 1

BEDROCK MONITORING WELL SHEET

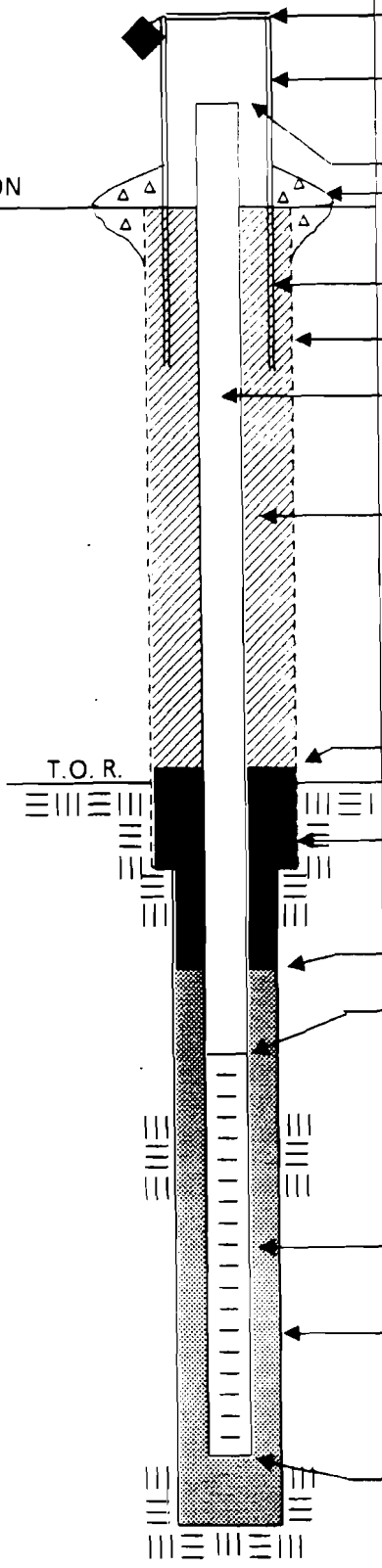
BORING NO.: _____

Tetra Tech NUS, Inc.

BEDROCK
MONITORING WELL SHEET
WELL INSTALLED IN BEDROCK

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____



ELEVATION OF TOP OF SURFACE CASING: _____

STICK UP OF CASING ABOVE GROUND SURFACE: _____

ELEVATION TOP OF RISER: _____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____

DIAMETER OF HOLE: _____

RISER PIPE I.D.: _____

TYPE OF RISER PIPE: _____

TYPE OF BACKFILL: _____

ELEVATION / DEPTH TOP OF SEAL: ____/____

ELEVATION / DEPTH TOP OF BEDROCK: ____/____

TYPE OF SEAL: _____

ELEVATION / DEPTH TOP OF SAND: ____/____

ELEVATION / DEPTH TOP OF SCREEN: ____/____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. SCREEN: _____

TYPE OF SAND PACK: _____

DIAMETER OF HOLE IN BEDROCK:

CORE / REAM: _____

ELEVATION / DEPTH BOTTOM SCREEN: ____/____

ELEVATION / DEPTH BOTTOM OF HOLE: ____/____

ATTACHMENT 2

OVERBURDEN MONITORING WELL SHEET

BORING NO.: _____

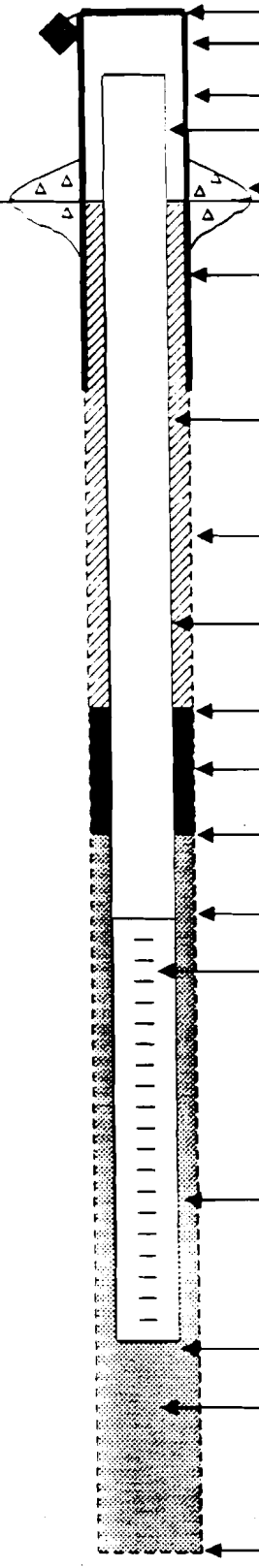
Tetra Tech NUS, Inc.

OVERBURDEN
MONITORING WELL SHEET

PROJECT _____ LOCATION _____
PROJECT NO. _____ BORING _____
ELEVATION _____ DATE _____
FIELD GEOLOGIST _____

DRILLER _____
DRILLING METHOD _____
DEVELOPMENT METHOD _____

GROUND ELEVATION



ELEVATION OF TOP OF SURFACE CASING : _____
ELEVATION OF TOP OF RISER PIPE: _____

STICK - UP TOP OF SURFACE CASING _____
STICK - UP RISER PIPE : _____

TYPE OF SURFACE SEAL: _____

I.D. OF SURFACE CASING: _____
TYPE OF SURFACE CASING: _____

RISER PIPE I.D. _____
TYPE OF RISER PIPE: _____

BOREHOLE DIAMETER: _____

TYPE OF BACKFILL: _____

ELEVATION / DEPTH TOP OF SEAL: _____ / _____

TYPE OF SEAL: _____

DEPTH TOP OF SAND PACK: _____

ELEVATION / DEPTH TOP OF SCREEN: _____ / _____

TYPE OF SCREEN: _____

SLOT SIZE x LENGTH: _____

I.D. OF SCREEN: _____

TYPE OF SAND PACK: _____

ELEVATION / DEPTH BOTTOM OF SCREEN: _____ / _____

ELEVATION / DEPTH BOTTOM OF SAND PACK: _____ / _____
TYPE OF BACKFILL BELOW OBSERVATION WELL: _____

ELEVATION / DEPTH OF HOLE: _____ / _____

STANDARD OPERATING PROCEDURE
NUMBER CTO166-13

PACKER TESTING OF UNCASED BOREHOLES

1.0 SCOPE AND APPLICATION

This Standard Operating Procedure (SOP) delineates protocols for performing packer tests in open holes in bedrock at the NSWC Crane facility. These tests will allow hydraulic properties of rock to be determined and ground water samples to be collected, if needed, from specific depth intervals.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Submersible pump with 0.5 to 10 gpm capacity
Inflatable packers
Drill rig and associated equipment
Teflon-coated drop tube
Stainless-steel cable
Pressure transducer and data recorder
Photoionization detector (PID)
Water-quality meter
High-pressure steel tubing

3.0 PROCEDURE

3.1 Determine depth intervals of the open borehole to be tested.

3.2 Lower the decontaminated packer assembly down to the first desired interval to be tested.

3.3 Pack off the top and bottom of the test interval with inflatable packers that are attached to high-pressure steel tubing.

3.4 Monitor the hydraulic pressure in the test section until the pressure reaches a constant value (i.e., steady state). Record times and pressure values on the data logger.

3.5 Once the pressure has reached steady state, turn on the pump, which will produce water at a rate of about 1 gpm.

3.6 Monitor the hydraulic pressure during pumping. Do not let the water level drop below the top packer elevation (i.e., do not let the packed interval become dewatered).

3.7 If the water level drops quickly, reduce the pumping rate to the lowest steady level and continue to record the water pressure.

3.8 Monitor water-quality parameters (pH, ORP, temperature, D.O., turbidity, and specific conductance) in accordance with SOP CTO166-16 in-line in the pump discharge line during the test. Record values every 2 to 3 minutes.

3.9 Monitor the flow rates during the test and record every 2 minutes. Any time the pump rate is intentionally reduced, record this information.

3.10 After approximately 15 minutes of pumping and when the ground water elevation has stabilized and is still well above the top packer elevation, a step increase in pumping rate may be achievable. Increase the pumping rate to about 5 gpm and repeat steps 3.6 through 3.9.

3.11 If, after about 15 minutes, the ground water elevation has stabilized, attempt to increase the pumping rate to 10 gpm. **Do not let the water level drop below the top of the packer assembly.** If the water pressure drops quickly, cut the pumping rate down to 7 or 8 gpm, or stop pumping altogether.

3.12 Record water pressures and water-quality parameters every 2 to 3 minutes. When the pressure transducer readings have stabilized (i.e., remained constant over a 5-minute period), the test is completed.

3.13 Turn off the pump and deflate the packers.

3.14 Lower the pump and the packer assembly to the next interval selected for testing.

3.15 Repeat steps 3.3 through 3.13 for next test interval.

3.16 When testing is completed, remove the packer assembly, pump, and other equipment from the hole. Decontaminate all equipment as described in SOP CTO166-17.

3.17 Contain all water produced during testing. Transport the IDW water to the central storage area in accordance with SOP CTO166-15.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-14

CALIBRATION AND CARE OF WATER QUALITY METER

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for the calibration and maintenance of field instruments used to measure water quality and for the proper documentation of calibration and maintenance. The YSI 6-Series Environmental Monitoring System will be used to measure pH, temperature, oxidation-reduction potential (ORP), specific conductance (SC), dissolved oxygen (DO), and turbidity in water. The YSI meter has a multiprobe sensor that can be used in conjunction with a flow-through cell attached to a pump discharge tube to measure water-quality parameters in a ground water discharge or can be immersed in a surface water body such as a stream, pond, or drainage ditch.

2.0 FIELD FORMS AND EQUIPMENT LIST

The following logbooks, forms, equipment, and supplies are required:

Site logbook
Equipment calibration log sheet
YSI Model 610-D and Sonde: multi-parameter water-quality meter with flow through cell
Equipment manual
Calibration kit
Deionized water, paper towels, spray bottle, etc.
Disposable medical-grade gloves (e.g., latex, nitrile)

3.0 PROCEDURES

This section describes the calibration procedure for the YSI Model 610-D. The meter is supplied with an instruction manual. Sections of this manual are reproduced in this SOP. The manual will be on site and will be used as the calibration guidance document for the meter's calibration (page 29, Section 2.6 of the

manual starts the calibration procedure). This procedure will list requirements for frequency of calibration and checks to be performed on the meter.

The YSI Model 610-D and Sonde is a multi-parameter, water-quality meter that may be used to measure open water bodies (streams, ponds, springs, etc.) with the probe guard installed. With the flow through cell attached, the meter has the ability to measure water-quality parameters in ground water via a pump discharge line. By performing the measurements in the discharge line coming directly from the well, the parameters are measured before the ground water comes in contact with the atmosphere. The parameters measured by the YSI for this field effort are as follows:

- DO
- SC
- Temperature
- pH
- ORP
- Turbidity

3.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

3.2 Calibration

All the parameters listed in Section 3.0 must be calibrated prior to the start of each field effort. After this initial calibration, the YSI will be checked each day that it is used. If the check shows any out-of-specification readings, the specific probe will be recalibrated. Meter specifications can be found in the equipment manual, starting on page 248. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration buffers and standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

3.3 Tips for Good Calibration

- The DO calibration is a water-saturated air calibration. Make certain to loosen the calibration cup seal to allow pressure to equilibrate before calibrating.

- Make certain that sensors are completely submersed in solution and readings are stable when calibration values are entered.

- Use a small amount of calibration solution (previously used solution may be used, then discarded for this purpose) to pre-rinse the sonde.

- Fill a bucket with ambient temperature water to rinse the sonde between calibration solutions.

- Make sure to rinse and dry the probe between calibration solutions. This will reduce carry-over contamination and increase the accuracy of the calibration.

4.0 MAINTENANCE

The YSI Meter will be rented for the duration of each brief field effort. Therefore, little field maintenance will be required. For any maintenance other than the routine cleaning, calibrating, or battery charging, the instrument should be returned to the vendor and a replacement sent immediately to the job site.

4.1 Meter Storage

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. During these breaks, the meter will be charged. One-half inch of tap or distilled water will be placed in the meter calibration cup and the cup threaded onto the sonde. The key for short-term storage of probes is to use a minimal amount of water so the calibration cup will remain at 100 percent humidity. The water level must be low enough so that none of the probes are actually immersed. Proper storage of the sonde between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

Multi-parameter short term storage key points:

- Use enough water to provide humidity but not enough to cover the probe surfaces.
- Make sure the storage vessel is sealed to minimize evaporation.

- Check periodically to make certain that water is still present.

4.2 Probe Cleaning

- Rinse the probe thoroughly with potable water.
- Wash the probe in a mild solution of Liquinox and water and wipe with paper towels and/or cotton swabs.
- Rinse and soak the probe in deionized water.
- If stronger cleaning is required, consult Section 2.10 on page 89 of the equipment manual.

Note: Reagents that are used to calibrate and check the YSI may be hazardous. Review the health and safety plan, Appendix A of the equipment manual, and Material Safety Data Sheets (MSDSs), all of which are on file in the field trailer.

5.0 ATTACHMENTS

1. Equipment Calibration Log

ATTACHMENT 1

EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

Date of Calibration	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot No.)	Remarks and Comments
			Pre-calibration	Post-calibration	Pre-calibration	Post-calibration		

STANDARD OPERATING PROCEDURE
NUMBER CTO166-15

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at the NSWC Crane facility. The following types of IDW will be generated during this investigation:

- Excess soil and rock materials remaining from subsurface drilling activities
- Well development water and purge water related to monitoring well installation and sampling
- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)
Decontamination equipment
Field logbook and indelible ink pen
Plastic sheeting and/or tarps
55-gallon drums with sealable lids
IDW labels for drums
Wastewater container tanks
Plastic garbage bags

3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities.

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include well development water, well purge water (collected during low-flow sampling), and decontamination solutions from drilling and sampling equipment. As they are collected, these wastewaters will be placed in a 300-gallon (or smaller) portable tank attached to a truck. Whenever the portable tank approaches full, it will be transported to a central location at NSWC Crane (adjacent to a Crane-designated sanitary sewer manhole). The water in the portable tank will be discharged to the sewer by gravity draining.

An accurate record will be kept by the FOL of all wastewaters that have been placed in the large holding tank. At a minimum, this information will include the following:

- The location and type of each water that has been placed in the tank (e.g., purge water from wells)
- The quantity of water from each source
- The date the wastewater was generated
- The date and time the wastewater was placed in the tank
- The person(s) present when the wastewaters were discharged to the sewer

3.2 Drill Cuttings and Cores

As cuttings are removed from borings, they will be screened for VOCs. If these results do not show above-background levels of VOCs, the cuttings materials will be mixed with bentonite and returned to the borehole when sampling activities in the hole are completed or spread on the ground in the area of the borehole. The backfill materials will be tamped as they are placed in the hole to increase density and reduce permeability of the backfill material.

If any soil materials from a screened borehole interval show evidence of contamination (based on the field screening results), then the soil material from the screened interval will be placed in a plastic trash bag (or directly in a drum if a larger quantity) and the bag will be tagged. Information included on the tag will consist of the hole from which the material came, the depth interval from which the material came, the date, and the name of the person filling out the tag. The bag will then be placed in a 55-gallon drum and sealed (more than one bag may be placed in a drum). The waste drums will be stored on site temporarily until laboratory results have been received concerning the soil samples that were collected from the suspect borehole. If the results indicate that no contamination is present in the soil samples, then the soils will be disposed on site. If the levels of contamination of any of the samples from a borehole exceed Toxicity Characteristic Leaching Procedure (TCLP) limits (using the total soil concentrations), then all

excess soil from the borehole will be considered as RCRA-hazardous and disposed off site in accordance with RCRA waste disposal regulations. Alternatively, the toxicity characteristic leaching procedure may be conducted for any constituent that could exceed the TC limits based on total concentrations.

Cuttings from well borings will be handled in the same manner as soil boring cuttings.

3.3 PPE, Pump Discharge Tubing, Direct-Push Technology (DPT) Sample Liners, and Incidental Trash

All PPE wastes, pump discharge tubes, DPT sample liners, and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NSWC Crane.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-16

LOW-FLOW WELL PURGING AND STABILIZATION

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for well purging and stabilization utilizing low-flow techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for low-flow purging.

Low-Flow Purge Data Sheet: A copy of this form is attached at the end of this SOP.
Ground Water Sample Log Sheet: A copy of this form and instructions for its completion are included in SOP CTO166-05.
Bound field logbook
Writing utensil
Photoionization detector (PID): The procedures for the operation of the PID are found in the health and safety plan and SOP CTO166-06.
Well key
Electronic water-level indicator: The water-level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01 foot (see SOP CTO166-18).
Electronic Programmable Controller, model 400: This controller regulates air flow in a bladder pump.
Cylinder of compressed nitrogen with regulator: Compressed gas serves as the power source for the bladder pump.
Multiple parameter water-quality meter: This unit measures and displays field parameters measured in the field including pH, dissolved oxygen, oxidation-reduction potential (ORP), temperature, and specific conductance (see SOP CTO166-14).
Flow-through cell adapter for water-quality meter
LaMotte Turbidity Meter: Used to measure turbidity (see SOP CTO166-14).
Purge water containers
Graduated cylinder and stopwatch: Used to calculate flow rate.

Decontamination supplies: SOP CTO166-17 describes required decontamination supplies.

Disposable medical-grade gloves (e.g., latex, nitrile)

3.0 PROCEDURES FOR WELL PURGING

3.1 Prior to mobilizing to the site, clean, check for proper operation, and calibrate above equipment in accordance with manufacturer requirements as necessary.

3.2 Follow the steps outlined in SOP CTO166-18 to obtain a static water-level measurement of the well to be purged. Record the information on the Ground Water Sample Log Sheet and the Low-Flow Purge Data Sheet. Leave the water-level meter suspended in the well casing.

3.3 Calculate one well casing volume as follows:

1. Obtain the total depth of the well by measurement.

2. Using the static water level determined in Step 3.2 of this SOP and the total depth of the well, calculate the well casing volume using the following formula:

$$V = (0.163)(T)(r^2)$$

where:

V	=	Static casing volume of well (in gallons).
T	=	Vertical height of water column (linear feet of water).
0.163	=	A constant conversion factor that compensates for the conversion of the casing radius from inches to feet, the conversion of cubic feet to gallons, and pi.
r	=	Inside radius of the well casing (in inches).

Note: For wells of 1-inch radius (2-inch diameter), V = 0.163 gallons per foot of water column.

3.4 Connect the pump controller to the well pump air supply (at the well cap) by following the instructions in the pump control manual. The pump controller must be turned off when it is being connected.

3.5 Connect the nitrogen cylinder to the pump controller. The nitrogen cylinder valve must be closed and the regulator line pressure set at zero pounds per square inch (psi) when it is being connected.

3.6 Following the instructions found in the water-quality meter manual, connect the flow-through cell to the pump discharge line (at the well cap).

3.7 Place the discharge tubing from the flow-through cell to direct the purge water discharge into the graduated cylinder or purge water container.

3.8 Following the instructions in the pump controller manual, start pumping water from the well.

3.9 Start with the initial pump rate set at approximately 0.1 liters per minute. Use the graduated cylinder and stopwatch to measure the pumping rate. Adjust pumping rates as necessary to prevent drawdown from exceeding 0.3 foot during purging. If no drawdown is noted, the pump rate may be increased (to a max of 0.4 liters per minute) to expedite the purging and sampling event. The pump rate will be reduced if turbidity is greater than 10 NTUs after all other field parameters have stabilized. If ground water is drawn down below the top of the well screen, purging will cease and the well will be allowed to recover before purging continues. Slow recovering wells will be identified and purged at the beginning of the workday. If possible, samples will be collected from these wells within the same 8-hour workday and no later than 24 hours after the start of purging.

The time to sample any given well will vary greatly due to the many variables associated with low flow purging and sampling:

- Stabilization of parameters
- Possible drawdown
- Analytical changes from quarter to quarter
- Varying QA sample requirements from quarter to quarter
- Variable pump rates

Normally, the time from the start of purging to the end of sampling will be between 1 and 4 hours.

3.10 Measure the well water level using the water-level meter every 5 minutes. Record the well water level on the Low-Flow Purge Data Form (attached at the end of this SOP).

3.11 Every 5 to 10 minutes, record on the Low-Flow Purge Data Form the water-quality parameters (pH, specific conductance, temperature, turbidity, oxidation-reduction potential, and dissolved oxygen) measured by the water-quality meter and turbidity meter. If the cell needs to be cleaned during purging operations, continue pumping (allow the pump to discharge into a container) and disconnect the cell. Rinse the cell with distilled water. After cleaning is completed, reconnect the flow-through cell and continue purging. Document the cell cleaning on the Low-Flow Purge Data Form.

3.12 Measure the flow rate using a graduated cylinder. Remeasure the flow rate any time the pump rate is adjusted.

3.13 During purging, check for the presence of bubbles in the flow-through cell. The presence of bubbles is an indication that connections are not tight. If bubbles are observed, check for loose connections.

3.14 Stabilization is achieved and sampling can begin when a minimum of one casing volume has been removed and three consecutive readings, taken at 5- to 10-minute intervals, are within the following limits:

- pH ± 0.1 standard units
- Specific conduct ± 5%
- Temperature ± 5%
- Turbidity less than 10 NTUs
- Dissolved oxygen ± 10%

If the above conditions have still not been met after the well has been purged for 4 hours, purging will be considered complete and sampling can begin. Record the final well stabilization parameters from the Low-Flow Purge Data From onto the Ground Water Sample Log Form.

If there is a need to leave a well during purging, there are two options:

- One, if the sampler must move for 30 minutes or less but still has a clear line of sight to the well, the sampler may leave the pump running and watch the well from a distance until he or she is able to return to the well.

- Two, if for whatever reason, the sampler must stop purging for an extended period of time or a clear line of sight cannot be maintained, the pump and cell will be shut down. All equipment and supplies will be loaded into the sample vehicle, and the well will be secured before the sampler departs.

In both cases, the time purging was stopped and restarted will be noted on the Low-Flow Purge Data Form.

3.15 Rinse the flow-through cell, the water-quality meter probes, and the turbidity cell with analyte-free water and pack the cell and meters for transport.

4.0 ATTACHMENTS

1. Low-Flow Purge Data Sheet

ATTACHMENT 1

LOW-FLOW PURGE DATA SHEET

Tetra Tech NUS, Inc.

LOW FLOW PURGE DATA SHEET

Well No.: _____

PROJECT: _____ **DATE:** _____
PROJECT NUMBER: _____ **WEATHER:** _____
SITE: _____ **PERSONNEL:** _____

Well Screen Depth: _____ / _____ ft.
Initial Water Level: _____ @ _____ hrs.

Pump Type/Material: _____
Pump Intake Depth: _____

Tide Cycle: ☐ High @ _____ ☐ Low @ _____ ☐ Not Affected

Total Purge Volume=_____ **(gal / L)** **Total Purge Time=**_____ **(min)**

Time	Water Level feet below TOC	Volume mL	Flow Rate mL/min	Pump Settings	Temp ºC	pH	Sp Cond mS/cm	DO mg/L	Turbidity NTU	Salinity ppt	Eh mV	Comments

Water Quality Meter (S/N): _____ Notes: _____
Control Box Type (S/N): _____
Turbidimeter (S/N): _____

STANDARD OPERATING PROCEDURE
NUMBER CTO166-17

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Waterproof pens
Non-latex rubber or plastic gloves
Cotton gloves
Field logbook
Potable water
Deionized water
LiquiNox detergent
Brushes, spray bottles, paper towels, etc.
55-gallon drum or other container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has

accumulated on the sampling equipment, remove the residue with a isopropanol wash and repeat the Liquinox wash.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the field logbook.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP CTO166-15 and the QAPP.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-18

MEASUREMENT OF WATER LEVELS IN MONITORING WELLS

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes procedures for determining water levels in monitoring wells.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following equipment and field forms are required for determining water levels in monitoring wells.

Ground Water Level Measurement Form: A copy of the Ground Water Level Measurement Form is attached.
Bound field logbook
Photoionization detector (PID): Operation and calibration of the PID is discussed in SOP CTO166-06.
Well key
Writing utensil
Electronic water-level indicator: The water-level indicator must have a cable of sufficient length to reach the water surface and be capable of measurements of 0.01 foot.
Decontamination supplies: SOP CTO166-17 describes decontamination procedures including decontamination supplies.

3.0 WATER-LEVEL MEASUREMENT PROCEDURES

3.1 Check the operation of the electronic water-level indicator or interface meter.

3.2 Record the well identification (ID), date, and time (using military time) on the Ground Water-Level Measurement Form.

3.3 Unlock the well and remove the well cap.

3.4 Place the well cap on a clean piece of plastic.

3.5 Check the well for the presence of organic vapors in the 2-inch polyvinyl chloride (PVC) riser pipe as follows:

1. Calibration of the PID shall be done in accordance with the calibration procedures described in SOP CTO166-06. Calibration of the PID shall be done at the field office prior to entering the field.

2 Insert the PID sample inlet straw approximately 3 inches into the riser pipe.

3 Record the PID reading on the Ground Water Level Measurement Form. If the reading is less than concentrations specified in the site-specific HASP, proceed to step 3.6. If the reading is greater than the concentration specified in the HASP, measure the concentration in the breathing zone. If the concentration in the breathing zone is less than the concentration specified in the HASP, proceed to Step 3.6. If the reading is greater than the specified concentration, allow the riser pipe to ventilate for 10 minutes and repeat the measurement of breathing zone concentrations until the concentrations fall below the level specified in the HASP before proceeding to step 3.6.

3.6 Ensure that the water-level indicator probe has been decontaminated before use, in accordance with the procedures outlined in SOP CTO166-17.

3.7 Slowly lower the probe into the well riser pipe until an audible and/or visible signal is produced, indicating contact with the water surface.

3.8 Read the ground water-level measurement from the top of the inner casing at the surveyed reference point to the nearest 0.01 foot.

3.9 Record the water-level measurement on the Ground Water Level Measurement Form.

3.10 Wind the meter cable measuring tape back onto the spool.

3.11 Replace the well cap and lock.

3.12 Decontaminate the meter's probe and cable following the procedures outlined in SOP CTO166-17.

3.13 Containerize any decontamination fluids and PPE in accordance with the procedures described in SOP CTO166-15.

4.0 ATTACHMENTS

1. Ground Water Level Measurement Sheet

ATTACHMENT 1

GROUND WATER LEVEL MEASUREMENT SHEET

Tetra Tech NUS, Inc.

GROUNDWATER LEVEL MEASUREMENT SHEET

Project: _____ Site: _____

Project No.: _____ Personnel: _____

Temperature: _____ Date: _____

Precipitation: _____ Level Indicator Type: _____

Tide: _____ Serial Number: _____

Well/Piezometer Number	Time	(A) Elevation of Reference Point (feet)*	(B) Water Level Indicator Reading (feet)*	=(A)-(B) Groundwater Elevation (feet)*	Total Well Depth (feet)*	Tidally Influenced	Comments

* Measurements to the nearest 0.01 foot.

Page ___ of ___

STANDARD OPERATING PROCEDURE
NUMBER CTO166-19

SURFACE WATER SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting surface water samples at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Surface Water Sample Log Sheet: A copy of this form is attached at the end of this SOP.

Field logbook

Writing utensil

Multi-parameter water-quality meter: The water-quality meter is used for the measurement of dissolved oxygen, pH, specific conductance, temperature, oxidation-reduction potential, and turbidity (see SOP CTO166-14).

LaMotte Turbidity Meter: Used to measure turbidity in the field.

Disposable sample containers: Disposable sample containers are used to fill sample containers and transport sample(s) to a pump for filtering.

Labeled sample containers: Prelabeled, certified-clean sample containers will be provided by the laboratory that performs the analyses.

Sample tags: One tag is to be completed and attached to each sample container.

0.45-micron filter assembly: These are single-use filter cartridges used to filter samples scheduled for dissolved metals or dissolved thorium isotope analyses. The filters become investigation-derived waste (IDW) after one use.

Peristaltic pump

Silicon tubing

Ziploc-type plastic storage bags

Shipping containers (coolers)

Trip blank sample (if VOC samples are being collected)

Temperature blank

3.0 SURFACE WATER SAMPLING PROCEDURES

3.1 The same methods will be used to collect surface water and seep samples. Sampling will start at the downstream end of a stream and proceed to the farthest upstream location.

3.2 While standing downstream or from the bank, gently remove any floating leaves or twigs that may be present in a sample pool area in a manner that will not disturb the bottom sediment.

3.3 While standing downstream or from the bank, place the sample container in the water at the sampling location at a 45-degree angle and lower it to approximately half the sample pool depth. With the mouth of the container facing upstream, fill the container with water, being careful not to disturb the sediment.

3.4 All samples will be collected into certified-clean, pre-preserved bottles (if preservation is required for the analysis to be performed) supplied by the laboratory performing the analyses. Sample containers for volatile constituents (VOCs) must be completely filled so no headspace exists in the container. Other sample containers should <u>not</u> be filled completely; a small amount of air should be left at the top. Sample containers will be collected in the following sequence:

Volatile organic compounds (VOCs)
Other Organics
Total metals
Nitrate
Nitrite
Total suspended solids (TSS)
Dissolved metals

3.5 Record the date and time that the sample containers are filled on the Surface Water Sample Log Sheet, the sample labels, the sample tags, and the Chain-of-Custody Form.

3.6 After the sample label and sample tag are completed and checked, place the sample container into a ziploc-type plastic storage bag and place the plastic storage bag holding the sample container into a cooler containing ice.

3.7 Repeat steps 3.3 through 3.6 until all the sample bottles containing unfiltered samples have been filled.

3.8 Fill two 1-liter unpreserved polyethylene bottles. Use these bottles to transfer the sample for field filtering. Set up a peristaltic pump for filtering of the dissolved metals samples. Using new, clean, disposable silicone tubing and a 0.45-micron filter, place the intake tubing from the pump into the transfer bottle with the filter attached to the discharge end and start the pump. Pre-rinse the filter with approximately 50-mL of sample water prior to filling the sample containers.

3.9 Using the discharge from the filter cartridge, fill one 1-liter polyethylene sample bottle for dissolved metals. Repeat steps 3.8 and 3.9 for these sample containers.

3.10 Obtain measurements of dissolved oxygen, pH, specific conductance, temperature, turbidity, and oxidation-reduction potential using the multi-parameter water-quality meter and LaMotte Turbidity Meter (see SOP CTO166-14). Record the readings in the appropriate fields on the Surface Water Sample Log Sheet.

3.11 Estimate the flow rate of the stream or spring in accordance with SOP CTO166-21. This is an estimate only. Round the flow rate to the nearest 5 gallons and record this number on the Surface Water Sample Log Sheet.

3.12 Decontaminate all equipment and load the equipment and the sample cooler in the sample vehicle for transport.

4.0 ATTACHMENTS

1. Surface Water Sample Log Sheet

ATTACHMENT 1

SURFACE WATER SAMPLE LOG SHEET

SURFACE WATER SAMPLE LOG SHEET

Project Site Name: _____
Project No.: _____

[ ] Stream
[ ] Spring
[ ] Pond
[ ] Lake
[ ] Other: _____
[ ] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[ ] Low Concentration
[ ] High Concentration

SAMPLING DATA

Date: / Time:	Color	pH	S.C. mS/cm	Temp. °C	Turbidity NTU	DO mg/l	Salinity %	Eh mV
Depth: / Method:								

SAMPLE COLLECTION INFORMATION

Analysis	Preservative	Container Requirements	Collected

OBSERVATIONS / NOTES

MAP

Circle if Applicable:

MS/MSD

Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
NUMBER CTO166-20

SEDIMENT SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for sediment sampling at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for sediment sampling.

Sediment Sample Log Forms: A copy of this form is attached at the end of this SOP.
Bound field logbook
Tags for each sample container
Disposable plastic trowels
Survey stakes and flagging: Used to mark sampling locations after completion of sampling.
Labeled sample containers: See SOP CTO166-01 for sample identification procedures. Sample containers are certified clean by the laboratory supplying the containers.
Plastic storage bags
Shipping containers (containing ice)
Surgical gloves
Indelible marker
Chain-of-Custody Form

3.0 SEDIMENT SAMPLE LOCATION SELECTION

In general, sediments composed of fine-grained materials with greater surface area available for adsorption are more desirable for sample selection. The fined-grained materials may act as a sink or reservoir for adsorbing heavy metals and organic contaminants even if surface runoff concentrations are below detection limits. Therefore, it is important to locate the specific sampling points where the sediment has the greatest percentage of fine particles. The sampling personnel will determine specific sampling locations with these goals in mind.

3.0 SEDIMENT SAMPLING PROCEDURES

3.1 The sampler will wear clean, disposable, surgical gloves. Clear vegetative matter or debris, if present, from the sample location using a disposable sampling trowel or spoon. Use the trowel to dig up and homogenize the sediment in an 18-inch-diameter circular area that is 6 inches deep. Stir the sediment within the circular area; do not move the sediment outside the circle. Also, do not dig or stir sediment that is deeper than 6 inches below the ground surface, until the next depth interval is sampled.

3.2 Use the same trowel to scoop the homogenized sediment into the requisite labeled sample containers. All sample jars will be filled in the following sequence:

- Volatile organics
- Other organics
- TAL metals plus and Sn (total)
- Nitrate
- Nitrite
- TOC
- pH
- Geotechnical parameters

3.3 Record the sample time (using military time) on the Sediment Sample Log Form and sample container labels and tags. Record all other information required on the labels and tags as specified by SOP CTO166-01.

3.4 Secure the tag to the neck of the sample container using a wire tie.

3.5 Place the tagged sample container into a plastic storage bag and then place the plastic storage bag holding the sample container into a cooler containing ice.

3.6 Record date, sampling site, site conditions, location map, and other information (e.g., presence and flow rate of water in channel) on the Sediment Collection Log Sheet. Enter the sample information onto the Chain-of-Custody Form in accordance with SOP CTO166-03.

3.7 Using an indelible marker, write the sample identification on a survey stake, and drive the stake into the ground at the sample location. Tack a piece of brightly colored flagging to the stake. In addition, tie a piece of flagging to an overhead tree branch or other eye-level object to improve the ability to relocate the sampling site in the future.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1

SOIL AND SEDIMENT SAMPLE LOG SHEET

Tetra Tech NUS, Inc.

SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

GRAB SAMPLE DATA:

	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Date:			
Time:			
Method:			
Monitor Reading (ppm):			

COMPOSITE SAMPLE DATA:

Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				

SAMPLE COLLECTION INFORMATION:

Analysis	Container Requirements	Collected	Other

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
NUMBER CTO166-21

ESTIMATING FLOW IN SMALL STREAMS

1.0 PURPOSE

This Standard Operating Procedure (SOP) provides technical guidance on the measurement of stream channel cross-section and flow in a small stream. Stream flow measurements will be obtained to evaluate the migration potential of contaminants in a stream channel. This method is applicable for free-flowing streams with uniform flow and widths greater than 3 feet and depths greater than about 6 inches. Stream flow rates will be measured at the same time as water-level measurements are collected.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Measuring rod or yardstick
Measuring tape
Waders or rubber boots
Colored flagging tape
2x2 inch wooden stakes
Bound field logbook
Waterproof pen
Orange or apple
Surface Water Sample Log Sheet
Indelible marker
Stopwatch

3.0 PROCEDURES

3.1 Establish a transect across the stream, along a straight reach where the stream is relatively narrow and velocity across the stream is relatively uniform. Orient the transect perpendicular to the flow.

3.2 Mark both ends of the transect with colored plastic flagging tape. Tie the flagging to a tree branch or other elevated (about eye level) object. Drive wooden stakes into the ground at both ends of

transect and tack a short piece of brightly colored flagging to the stakes. Use a black marker to identify the transect number, which will be the same as the surface water sample location.

3.3 Measure the total distance across the stream with a measuring tape and record the stream width (in feet) in the field logbook.

3.4 Measure the depth of water in the stream at three or four points across the stream. Record these measurements and calculate an average depth. Record the average depth (in feet) in the field logbook.

3.5 Measure a distance of 20 feet upstream of the transect. Tie a piece of flagging on a tree branch or other visible location above the stream.

3.6 Go another 20 feet upstream and place the orange or apple in the center of the stream or where the water current is fastest.

3.7 Using a watch with a timer or a stopwatch, measure the length of time it takes the orange (or apple) to travel from the first marker flag to the transect location (distance of 20 feet). Record the time.

3.8 Repeat step 3.7 two more times. Record the readings and calculate an average time.

3.9 Calculate approximate flow rate in the stream channel by multiplying the stream width (from step 3.3) times the average depth (from step 3.4) times the average velocity (from step 3.8). Record the estimated flow rate (in ft^3/s) in the field logbook and on the Surface Water Sample Collection Log Sheet.

STANDARD OPERATING PROCEDURE
NUMBER CTO166-22

HYDRAULIC CONDUCTIVITY TESTING

1.0 PURPOSE

This Standard Operating Procedure (SOP) provides technical guidance for the performance and evaluation of an in-situ hydraulic conductivity testing (slug testing) in monitoring wells at the NSWC Crane facility.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Solid slug
Pressure transducers and data recorder, including instruction manual
Manual water-level indicator
Hydraulic Conductivity Testing Data Sheet
Watch
Decontamination equipment and supplies
Field logbook
Measuring tape

3.0 PROCEDURES

Slug tests are short-term tests designed to provide approximate hydraulic conductivity values for the portion of a formation immediately surrounding the screened/open interval of a well or boring. These tests are much less accurate than pumping tests, because a much more localized area is involved. Therefore, a number of slug tests are typically performed and averaged to determine a representative hydraulic conductivity value for the formation tested. Performance of slug tests may be preferable to pumping tests in situations where handling of large volumes of contaminated water is a concern or when time or budget constraints preclude the more expensive and time-consuming setup and performance of a pumping test.

The procedure is summarized below:

3.1 Determine the total depth of the well using a weighted tape or other measuring device. A pressure transducer attached to a data logger will be placed in the well approximately 1 foot from the bottom of the well. The transducer will be positioned so that it is about 5 to 10 feet lower than the slug.

3.2 Record the well number, the transducer probe identification number being used, the PSI rating for each probe, the depth below top of casing where each probe is positioned, the static water level in the well, and any other information relative to the setup and performance of the slug test. Data and information should be recorded in a bound field notebook and on the Pumping Test Data Sheet.

3.3 A falling-head test can be performed where the slug is lowered into the well and the rate of water-level fall with respect to time is recorded until equilibrium is reached. A rising-head test can be performed where a slug is lowered into the well and the water is allowed to equilibrate, then the slug is removed and the rate of water-level rise is measured with respect to time. Falling head slug tests should only be performed in wells with fully submerged screens, and rising head slug tests can be performed in wells with either partially or fully submerged screens or open intervals.

3.4 Remove or insert the slug and immediately start the data logger. Record the starting time for the data logger on the form sheet.

3.5 Manually measure the depth to water with a water depth indicator to estimate the rate of recovery while the data logger is in the sleep mode. Enter the reading onto the form sheet, along with the corresponding transducer reading from the same time.

3.6 Observe the water-level readings when the data logger can be read. Record the times and the readings on the form sheet.

3.7 Rate of recovery measurements shall be obtained from time zero (maximum change in water level) until water-level recovery exceeds 90 percent of the initial change in water level. In low-permeability formations, the test may be cut-off short of 90 percent recovery due to time constraints. Time intervals between water level readings will vary according to the rate of recovery of the well. For a moderately fast recovering well, water-level readings at 0, 0.1, 0.2, 0.3, 0.4, 0.5, 0.75, 1.0, 1.25, 1.5, 2.0, 2.5, 3.0, 4.0, . . . minutes may be required. With practice, readings at down to 0.05-minute (3 seconds) time intervals can be obtained with reasonable

accuracy, using a pressure transducer and hand-held readout. For wells that recover very fast, the pressure transducer and data logger can be set on a logarithmic recording interval. Time intervals between measurements can be extended for slow recovering wells. A typical schedule for measurements for a slow recovering well would be 0, 0.25, 0.5, 0.75, 1.0, 1.5, 2.0, 3.0, 4.0, 6.0, 8.0, 10.0, 15.0, 20.0, 30.0, . . . minutes from the beginning the test. Measurements will be taken from the top of the well casing.

3.8 Stop the test when equilibrium is reached and repeat as necessary to ensure reproducibility.

3.9 Remove the pressure transducer, the slug, and the cables from the well and thoroughly decontaminate, in accordance SOP CTO166-17.

3.10 Check all field notes, copy, and place into one file for each test. Download the data recorder as soon as possible and check data. Make an electronic file and paper file of all data and place with the file for evaluation later. Confirm that the data are usable for the intended analysis prior to leaving the field. Time and recovery should be field plotted on semilog graph paper to determine the data quality. The data set should plot along a sloped, straight line. If excessive data scatter is observed, the test should be rerun until acceptable results are obtained.

4.0 PERSONNEL

A qualified geologist or hydrogeologist, with experience with these test procedures and equipment, will be needed for each sampling team to carry out the hydraulic conductivity tests.

5.0 ATTACHMENTS

1. Hydraulic Conductivity Testing Data Sheet

ATTACHMENT 1

HYDRAULIC CONDUCTIVITY TESTING DATA SHEET

HYDRAULIC CONDUCTIVITY TESTING DATA SHEET

PROJECT NAME: ..... WELL/BORING NO.: .....

PROJECT NO.: ..... GEOLOGIST: .....

WELL DIAMETER: ..... SCREEN LENGTH/DEPTH: ..... TEST NO.: .....

STATIC WATER LEVEL (Depth/Elevation): ..... DATE: .....

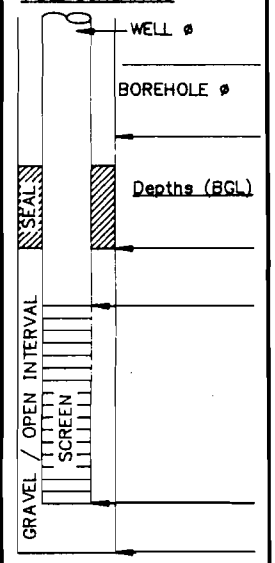
TEST TYPE (Rising/Falling/Constant Head): ..... CHECKED: .....

METHOD OF INDUCING WATER LEVEL CHANGE: ..... PAGE ..... OF .....

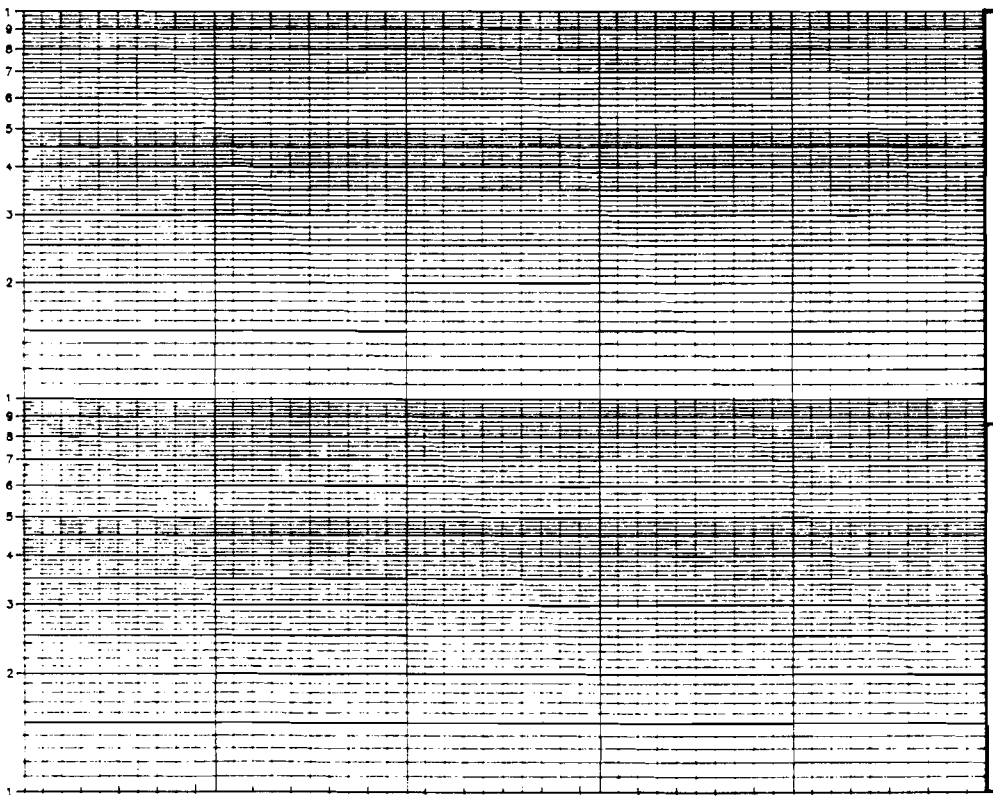
REFERENCE PT. FOR WL MEAS. (Top of Casing, Transducer, etc.): .....

ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD (ΔH) (feet)	ELAPSED TIME (min. or sec.)	MEASURED WATER LEVEL (feet)	DRAWDOWN OR HEAD (ΔH) (feet)

WELL SCHEMATIC



▽ Indicate SWL Depth on Drawing



REMARKS:

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CALCS,SKETCH MAPS, ETC.:

Quality Assurance Project Plan

for

Mine Fill A (SWMU 12)
Mine Fill B (SWMU 13)
Cast High Explosives Fill/
B146 Incinerator (SWMU 16) and
Pyrotechnic Test Area (SWMU 19)
RCRA Facility Investigation

Volume 2 - Appendix I

Naval Surface Warfare Center
Crane

Crane, Indiana

Southern Division
Naval Facilities Engineering Command

Contract Number N62467-94-D-0888

Contract Task Order 0166

July 2002

STANDARD OPERATING PROCEDURE SUBMITTALS

Laucks Testing Laboratory, Triangle Laboratory, and NSWC Crane Laboratory Analytical Standard Operating Procedures (SOPs) have previously been submitted as part of other QAPPs. Laucks Testing Laboratory and Triangle Laboratory SOPs were submitted as part of the approved Quality Assurance Project Plan for Ammunition Burning Grounds Little Sulphur Creek and Jeep Trail Resource Conservation and Recovery Act Phase III RCRA Facility Investigation. NSWC Crane Laboratory analytical SOPs were submitted as part of the approved Work Plan for RCRA Facility Investigation Corrective Measures Study and Risk Assessment at Solid Waste Management Unit 2-Dye Burial Grounds. All analytical SOPs utilized for analysis of samples collected at SWMUs 12, 13, 16, and 19 are listed. However, analytical SOPs which have not changed are incorporated by reference and only SOPs which have been changed are included in this submission and are shown in italics in the Appendix I Table of Contents.

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
TABLE OF CONTENTS

SOP No.	REV No.	QAPP/WP	TITLE
LAUCKS TESTING LABORATORIES			
LTL-1001	4	126[1]	Elements of SOP and Method Formats
LTL-1002	5	126[1]	Document Tracking and Control
LTL-1003	2	126[1]	Chain-of-Custody and Documentation Procedures
LTL-1004	4	126[1]	Documentation of Analyst Competence and Training
LTL-1005	5	126[1]	Analytical Balances
LTL-1006	3	126[1]	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
LTL-1007	3	126[1]	Use of Instrument Records and Logbooks
LTL-1008	6	126[1]	QC Corrective Action
LTL-1009	3	126[1]	Blind Spike Program
LTL-1011	4	126[1]	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits
LTL-1012	2	126[1]	Solvent QC Monitoring for Trace Residue Analysis
LTL-1013	4	126[1]	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
LTL-1017	1	126[1]	Internal Audit Procedures
LTL-1018	1	126[1]	Overview of Review and Approval Practices for Validatable Data Packages
LTL-1019	2	166[2]	Controlling, Maintaining, and Monitoring Laboratory Logbooks
LTL-1020	2	166[2]	Integration of IC, GC, HPLC and GCMS Peaks
LTL-2001	6	166[2]	Waste Segregation and Disposal
LTL-3000	12	166[2]	Extraction Method for Base, Acid and Neutral Compounds in Water (8270C by 3520C)
LTL-3001	4	166[2]	Extraction Method for Polynuclear Aromatics in Water (8270C by 3520C)
LTL-3011	2	166[2]	Extraction Method for Chlorinated Herbicides in Soil (8151A by 3550B)
LTL-3077	5	166[2]	Extraction Method for Ordnance Compounds in Water
LTL-3100	11	126[1]	Extraction Method for Base, Acid and Neutral Compounds in Soils (8270C by 3550B)
LTL-3161	5	166[2]	Extraction Method for Ordnance Compounds in Soils (8330)
LTL-3202	3	166[2]	Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081A/8082 by 3510C)
LTL-3302	2	126[1]	Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Soil (8081A/8082 by 3550B)

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LTL-3510	8	166[2]	Extraction Method for Chlorinated Herbicides in Water (8151A by 3510C)
LTL-3550	5	166[2]	Extraction Method for Picric and Picramic Acids in Water (3510C)
LTL-3555	4	166[2]	Extraction Method for Piciric and Picramic Acids in Soil (3550B)
LTL-4002	6	126[1]	Electronic Sample Entry and Log-In
LTL-4103	0	126[1]	Reviewing a Sample Delivery Group (SDG) and Updating Projects in the LIMS
LTL-4201	0	126[1]	Package Deliverables for all Reporting Levels
LTL-6006	0	166[2]	Cation –Exchange Capacity of Soils (Sodium Acetate) by SW 846 Method 9081
LTL-6008	0	166[2]	The Determination of Acid Volatile Sulfides and Simultaneously Extracted Metals in Sediment
LTL-7003	2	126[1]	Inorganic Glass Cleaning Procedure
LTL-7009	3	126[1]	Microwave Assisted Acid Digestion of Aqueous Samples and Extracts Using SW846 Method 3015
LTL-7010	3	166[2]	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy by SW846 Method 3010A
LTL-7012	1	126[1]	Microwave Assisted Acid Digestion of Sediments, Sludges, Soils, and Oils Using SW846 Method 3015
LTL-7015	1	126[1]	Acid Digestion of Sediments, Sludges, and Soils Using SW846 Method 3050B
LTL-7202	4	126[1]	Metals Analysis Using Inductively Coupled Plasma – Mass Spectrometry (ICP/MS) SW846 Method 6020
LTL-7501	2	166[2]	Operation of the PE FIMS 400 for the Analysis of Hg.
LTL-8000	2	126[1]	Determination of Retention Time Windows
LTL-8019	4	166[2]	Determination of Non-Halogenated Volatile Organics by SW 8015B
LTL-8084	5	166[2]	Analysis of Organochlorine Pesticides and PCBs by SW 846 Methods SW 8081A and 8082
LTL-8151	4	166[2]	Analysis of Chlorinated Herbicides by 8151A
LTI-8265	4	166[2]	Determination of Volatile Organic Compounds by Method 8260B
LTL-8277	2	166[2]	Determination of Polynuclear Aromatic Hydrocarbon Compounds by Selective Ion Monitoring (SIM) Method 8270C
LTL-8279	3	166[2]	Determination of Semivolatile Organic Compounds by Method 8270C and 8270D
LTL-8302	1	126[1]	HPLC Ordnance 8330 Data Review

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LTL-8330	11	166(2)	Determination of Nitroaromatics and Nitramines by SW-846 Method 8330
LTL-9104	3	166(2)	Preparation and Analysis of Total and Amenable Cyanide According to EPA Methods 335.1, 335.3, and SW 846 9012A
LTL-9110	8	166(2)	Determination of Anions by Ion Chromatography by Method 300.0A or 9056
LTL-9113	2	166(2)	pH in Soil and Waste Samples Using SW846 Method 9045C
LTL-9115	3	166(2)	The Determination of Total Organic Carbon in Water Samples by EPA415.1 and SW-846 9060
LTL-9116	6	166(2)	The Determination of Total Organic Carbon in Soil/Sediment Samples Using Modified SW-846 Method 9060
LTL-9136	0	166(2)	Determination of Perchlorate by Ion Chromatography by EPA Method 314.0
LTL-9202	3	166(2)	Total Suspended Solids (Non-Filterable Residue) Using EPA Method 160.2
TRIANGLE LABORATORY			
2.04	6	166(2)	Nonconformance
5.01	10	166(2)	Sample Receipt, Identification, Handling, and Storage
DSP 105	16	166(2)	Extraction of PCDD/PCDF from Solids (not tissue) – 8290
DSP 161	18	166(2)	Extraction of PCDD/PCDF from Water for Methods 1613, 8290, and 551
DHR 182	9	166(2)	PCDDs and PCDFs by HRGC/HRMS – Method 8290
DPA 104	12	166(2)	Data Package Assembly and Shipping
NSWC CRANE LABORATORY			
CR4052-PD-0001	1	166(2)	Quality Assurance and Control Plan
CR4052-PD-0100	3	166(2)	Document Control
CR4052-PD-0150	4	166(2)	Record Retention
CR4052-PD-0200	3	166(2)	Preventive Maintenance
CR4052-PD-0220	0	166(2)	Chemical/Supply Management
CR4052-PD-0250	1	166(2)	Calibration
CR4052-PD-0300	0	166(2)	Training Overview
CR4052-PD-0450	0	166(2)	Performance Evaluation
CR4052-PD-0500	1	166(2)	Chain of Custody: Soil and Water Samples
CR4052-PD-0600	2	166(2)	Customer Reports

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SOP No.	REV No.	QAPP/WP	TITLE
CR4052-PD-0700	0	166[2]	Glassware Cleaning Procedure
CR4052-PD-2500	0	010[3]	Determination of Organic Colorants in Environmental Matrices by HPLC

Shading indicates SOPs that have been revised or that have not been included in previous QAPPs.

1 "Quality Assurance Project Plan for Ammunition Burning Grounds Little Sulphur Creek and Jeep Trail Resource Conservation and Recovery Act Phase III RCRA Facility Investigation," NSWC Crane, April 2001.

2 "Quality Assurance Project Plan for Mine Fill A (SWMU 12), Mine Fill B (SWMU 13), Cast High Explosives Fill/B146 Incinerator (SWMU 16), and Pyrotechnic Test Area (SWMU 19) RCRA FRI Investigation," NSWC Crane, February 2002.

3 Risk Assessment Work Plan for Risk Assessment at SWMUs 4, 5, 9, and 10," August 2000.

SOP No.—standard operating procedure number.
REV No.—SOP revision number.
QAPP—quality assurance project plan.
WP—work plan.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1019

Title: **Controlling, Maintaining, and Monitoring Laboratory Logbooks**

Revision history:

Number	Date
0	4/11/96
1	5/13/98
2	11/17/00

Written by: Harry Romberg Date: 11-22-00
Harry Romberg, QA Officer

Approved by: Kathy E Kreps Date: 11-22-00
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 Scope

1.1.1 The maintenance of logbooks is essential to monitoring all aspects of laboratory operations including instrument and method performance and in tracking analyses. It is also important in confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. This document applies to all personnel involved in the preparation, control and use of laboratory notebooks.

1.1.2 More specific instructions for maintaining logbooks can be found in pertinent SOPs, such as LTL-1007 "Maintaining Instrument Records and Logbooks" or LTL-1005 "Analytical Balances" or others specific to other laboratory operations.

1.2 Purpose

1.2.1 The purpose of this SOP is to define the practices used to maintain control and use of laboratory logbooks. This SOP is not intended as a specific description of any particular logbook type but covers the practices that must be in place for all logbooks employed at Laucks.

1.3 Definition of Terms

1.3.1 Logbook - Any bound or unbound document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs, standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs, or any other regularly maintained record of activity.

2. Requirements

2.1 Control

2.1.1 Documents that specify quality requirements or activities affecting quality or evidentiary activities shall be controlled to ensure that correct documents are being used and properly archived when completed. This insures that logbooks can be found at later dates rather than lost or prematurely disposed.

2.2 Maintenance

2.2.1 Quality assurance records (logbooks) shall be compiled and maintained in accordance with approved procedures.

2.3 Monitoring

2.3.1 Logbooks should be periodically monitored to ensure they are being properly maintained and information is being correctly recorded. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Pages from these logbooks, however, should be at least checked for accuracy and completeness as the individual data packages are reviewed. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. At any time, whether during routine data review or specific logbook monitoring, if discrepancies are found, they should be corrected not only on the copies of logbook pages which may accompany a data package but on the original logbook page(s). If more systematic problems are found, the originator of the discrepancies must be re-trained in the proper documentation technique.

3. Responsibilities

3.1 Quality Assurance Manager

- Maintain the logs for control of laboratory notebooks and provide control numbers and labels as required.
- Approve format and proposed content of laboratory notebooks; minor changes to pre-printed forms do not need QA approval as long as their basic content does not change.
- Maintain master copies of notebook pages (in instances where pre-printed pages with a specific format are used); this may be in electronic or hardcopy form or both.
- Monitor satisfactory implementation of the requirements of this SOP

3.2 Responsible Supervisor

- Determine the format and content of notebooks used in their respective areas.
- Ensure that QA has been provided with an electronic version of all pre-printed logbook pages in order that they are later available for reprinting or editing.
- In coordination with QA and the respective analyst(s) ensure that all laboratory notebooks are properly labeled, including the appropriate control number.
- Ensure that personnel are adequately trained in the proper use of laboratory notebooks

- Periodically review laboratory notebooks to verify satisfactory implementation of the requirements of this SOP. Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

3.3 Analyst

- Ensure that they are using the appropriate logbook and understand how to properly fill in the required fields. In general, this not only means entering correct information but that no pertinent information is left out or not entered in the assigned fields.

- Ensure that any new logbook has been given a logbook number by QA before beginning to use it.

- Ensure that the logbook is clearly identified with an instrument ID and purpose or other appropriate title which will enable the analyst to easily identify the logbook.

- Ensure that if pre-printed logbook pages need to be modified, the modifications are approved by their supervisor and that an electronic copy or, if requested an original hardcopy have been provided to QA.

4. Procedure

4.1 Notebook Structure

4.1.1 Laboratory notebooks may be either bound or unbound as described below. Most logbooks should be bound in some fashion but it is recognized that this is not always possible, such as for vendor service records. These records may be stored in 3-ring binders or other suitable notebooks but they may **NOT** be stored loose-leaf or in a simple manila folder.

4.1.2 In some instances, logbooks may be created from instrument printouts or other pages that do not lend themselves to being pre-bound. In these instances, the log sheets may be stored in a 3-ring binder or other storage until enough sheets have been accumulated to have them bound with the laboratory comb binder.

4.1.3 All logbooks whether bound or unbound must be controlled by QA as designated by the appropriate QA Book Number label (see example in Appendix 1).

4.1.4 Bound notebooks shall conform to the following:

- Where feasible, binding will be of a type that will make the removal and reinsertion of pages readily noticeable.
- If pre-printed and bound, all pages will be sequentially pre-numbered. If the format of the notebook permits the use of the reverse side of the pages, both sides of each page will contain a sequential page number.
- Each page of the pre-printed bound notebook will contain, as a minimum, the laboratory name, logbook title, and sequential page number. Other elements may also be necessary for any specific logbook.

4.1.5 Unbound notebooks shall conform to the following:

- Unbound pages will be contained in a binder or folder that prevents loss and provides protection from damage.
- Each unbound page will contain a unique identifier (e.g., run number/date). For identification purposes, a continuous printout on fanfold computer paper requires only one identifier unless the sheets are separated.
- As noted above in 4.1.2, some unbound logbooks may eventually be bound if practical.

4.1.6 All notebooks will contain the following information on the cover:

- Laboratory name, Laucks Testing Laboratories, Inc.
- Control number assigned by the Quality Assurance Officer
- The department to which the logbook was issued
- The use of the logbook (i.e. balance calibration, instrument run-log, etc.)
- The department book number or title uniquely identifying that book, as required to identify the specific use of the book. This may include an instrument number or other logbook ID (such as a standards logbook ID). This is in addition to the QA logbook number.
- Start Date, the date on which the first entry was made
- End date, the date on which the last entry was made

4.2 Control of Logbooks

4.2.1 The QA Officer will maintain a master log of laboratory notebooks that contains as a minimum, the following information:

- Unique control number for each logbook
- Logbook title, which should reflect the type of information to be entered.
- Department to whom issued, for accountability only. A logbook will generally be assigned to a work station or function, and in no way is a laboratory notebook to be considered a "personal" notebook.
- Date issued, for accountability only.
- Date closed, for accountability.

4.2.2 Master sheets for each logbook will be maintained by the QA Officer in cooperation with the responsible supervisor and analysts and will be utilized for producing notebooks when required.

4.3 Use of Laboratory Logbooks

4.3.1 The notebook is the basic document for recording information. Entries should be made into the notebook in real time, not written on scratch paper and transferred later.

4.3.2 Handwritten entries should be legible and entered in black or blue indelible ink.

4.3.3 Computer-generated data should be printed out and collected at appropriate times to represent the activities being recorded.

- Computer printouts may be either placed in unbound notebooks as described above, or inserted into bound notebooks.
- Computer printouts or other material inserted into bound notebooks must be securely fastened (tape is preferred) in such a way that removal and insertion of material can be determined readily.

4.3.4 When information from related activities is recorded in more than one notebook, provide adequate cross-reference information in all affected notebooks so that all pertinent data can be readily accessed.

4.3.5 Do not skip pages when entering data. For example, if data is not readily available for entry, do not leave space for later entry. Enter the data when it becomes available and provide adequate cross-references if required.

4.3.6 In cases where partial or complete pages must be left blank and not used, indicate the unused portion by placing a horizontal line at the beginning and end of the unused portion and connecting opposite ends of the horizontal lines with a diagonal, resulting in a Z-shaped figure. The individual striking out the blank area will initial and date the diagonal.

4.3.7 Errors or other changes must be deleted in a similar fashion or with a single-line cross-out which has been initialed and dated. **No erasures, overwriting, white-out or multiple-line cross-outs (blacking out) are acceptable.**

4.3.8 When pre-printed formats are used and all possible entries are not required, the remaining blanks may be struck out with a Z as described above, or entries such as N/A may be placed in the unused blanks.

4.3.9 The individual entering information into the notebook shall initial and date each page used, or in the case of logbooks with ongoing records which do not occupy the entire page, such as maintenance logs or balance logs, each individual entry.

4.4 Supervisory Monitoring of Laboratory Logbooks

4.4.1 Standard logbooks and run logs should be monitored at least semiannually by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

4.4.2 Logbooks should be reviewed using the review items provided in Appendix II, although it is not necessary to actually document the review using this checklist.

4.4.3 Errors should be formally brought to the attention of the responsible individual through the use of Corrective Action Forms. If errors are correctable or items can be corrected for legibility problems, they should be corrected using the proper error correction technique.

4.4.4 Logbooks that have been reviewed are marked with a fluorescent yellow or other colorful label that looks similar to the label in Appendix III.

4.5 QA Monitoring of Laboratory Logbooks

4.5.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained. This will generally be done approximately annually as part of routine audits. This observation does not preclude the requirement for supervisory review.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.: _____

Issued To: _____

Used For: _____

Dept. Book No.: _____

Start Date: _____

End Date: _____

Appendix II

Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced before it completely falls apart.

Appendix III

Example Logbook Review Label

Logbook pages _____ through _____
have been reviewed for completeness and
spot-checked for accuracy.

Initials:_____ Date:_____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1020

Title: **The Integration of IC, GC, HPLC, and GC/MS Peaks**

Revision history:

Number	Date
0	08/26/96
1	08/21/97
2	05/02/02

Revised by: Monica Carr, Organics Division Manger Date: 5/9/02

Reviewed by: Harry Romberg, QA Officer Date: 5-9-02

Approved by: Kathy Kreps, Laboratory Director Date: 5/9/02

Controlled Document

No. 20 Assigned to: TeTr

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1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the integration process for chromatographic data, the procedures for manual integration, and the procedures for documenting manual integration.

1.1.2 Integration identifies peaks found in the data collected during data acquisition and characterizes them. The software uses the integrated peaks to determine the identity and quantity of compounds in the samples. The peak area, peak height, peak type, baseline, and retention time of each peak in a chromatogram are determined by integration. It is expected that situations will arise when the automated quantitation provide inappropriate quantitations. This normally occurs when there is compound co-elution, baseline noise, or matrix interference. Some peaks, due to limitations of the software, will need to be manually integrated. The manual integration process must be documented as outlined in section 5 of this SOP.

1.1.3 Manual integrations have been a source of serious concerns throughout the laboratory industry as a tool of fraud. There have been labs as well as individuals who have been penalized due to improper manual integration, or inadequate documentation that their actions were appropriate. Therefore, it is imperative that proper and honest and completely documented procedures are followed.

1.1.4 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Definition of Terms

1.2.1 Manual Integration - any intervention by an analyst or supervisor to change the peak area, peak height, baseline, peak type, or retention time of a chromatographic peak.

2. Software

2.1.1 GC Acquisition-HP/EZChrom
2.1.2 GC/MS-Teknivent/EnviroQuant/Chemstation
2.1.3 GC-Target

3. Responsibilities

3.1 Analyst

3.1.1 The analyst is responsible for reading and understanding this SOP and that which is applicable to the method of analysis. The analyst must also perform and document all manual integrations as specified in this SOP.

3.2 Supervisor

3.2.1 The supervisor or designated data reviewer must verify that all manual integrations are performed according to this SOP. The supervisor or designated data reviewer must document that this verification has occurred per the applicable Data Review, Validation, and Reporting SOP.

4. Operation Procedures

4.1 Integrator operation

4.1.1 The ideal chromatogram has perfectly symmetric peaks separated from each other with periodic baseline points. It is common to encounter split peaks, deformed peaks, merged peaks, sloping baselines, noise, spikes, shoulders on peaks, and a host of other calamities.

4.1.2 To maximize the chances of obtaining ideal chromatograms, first optimize the chromatography.

4.1.3 Peak recognition and integration sequence - As the integrator scans the data, it examines the slope (vertical distance between points) and curvature (positive or negative). So long as these remain within preset bounds data is interpreted as the baseline. If the bounds are exceeded, a peak may be starting. If the condition persists, the integrator decides that it is on the upslope of a peak.

4.1.4 The curvature changes to negative about halfway up the peak. This is the inflection point where the peak starts to round over approaching the apex. Passing the top, the slope becomes negative and the integrator is on the downslope. Another inflection point comes on the downslope and finally the peak returns to the baseline (Figure 1).

4.1.5 Finite width of integration slices - The slope changes from positive to negative at the top of the peak. However for area slices having finite width the integrator can only determine which slice contains the peak apex. To get better values for the retention time and peak height, the

integrator takes the slice containing the-apex and one slice on either side, fits them to a quadratic equation, and solves the equation to find the highest point (Figure 2).

4.1.6 Optimizing peak recognition - The best conditions for recognizing isolated symmetric peaks on a quiet baseline is to match the peak width parameter to the measured width of the peaks at half height. Threshold should be a few units less than the highest value still capable of detecting the peak. When peaks cluster together or the baseline slopes or is noisy, these ideal values must be modified. Figure 3 shows the effects of changing the values.

4.1.7 Manual integration - It is important that the analyst is familiar with the compounds that are routinely analyzed. Knowing the response and peak shape of the standard is important for consistency in integration. It is best to optimize the method to process data so that manual intervention is minimized and peak integration is more consistent. If manual integration is performed, it is important to be consistent for a given analyte in the standards, blanks, spikes, and samples.

4.1.8 Allowable manual integrations - Some common reasons for manual integration are:

4.1.8.1 Split peaks (attachment 1)

4.1.8.2 Tailing (attachment 2)

4.1.8.3 Retention time shifts (attachment 3)

4.1.8.4 Mis-identification (attachment 3)

4.1.8.5 Merged peaks (attachment 3)

4.1.8.6 Secondary ions or qualifier ions (attachment 4)

4.1.8.7 Baseline shifts (attachment 5)

4.1.8.8 Skimming versus dropped baseline (attachment 6)

4.1.9 Improper manual integrations - These practices are not allowed and warnings up to and including termination of employment will follow any documented cases of improper manual integration. If you are unsure about a manual integration ask your supervisor or QA.

4.1.9.1 Adding area by including other peaks. (attachment 7)

4.1.9.2 Improper baseline - this includes the practice of having the baseline moved up the side of a peak to decrease the area. (attachment 8)

4.1.9.3 Changing a proper integration to make the peak "in".

4.1.10 Special rules for fuel analyses (*e.g.*, gasoline, diesel) - The integration of these multi-component analytes requires special integration rules. The area of all peaks (with the exception of the surrogates) and the area of the non-resolved components (hump) are grouped together to determine the quantity of the analyte. The baseline is fixed at the start and held at a constant level for the entire run. When integration of the baseline for fuel analyses is performed, the quantitation report will not be dated and initialed by the analyst. Instead an explanation of the baseline integration will be documented on the quantitation report or in the appropriate SOP and discussed in the sample narrative. Some acceptable multi-analyte integrations are shown in attachment 9.

5. Documentation

5.1.1 When any manual integration is performed, a graphic copy of the peak with the integration marks is generated and put into the folder with the chromatograms and quantitation reports for that sample. The three different software systems used in the laboratory are listed below with the commands for generating the copy. On the quantitation report the analyst must initial, date, and give a brief description of the reason for the manual integration (table 1). In addition, the analyst must provide chromatograms from both before and after the manual integration. The manual integration must also be documented in the associated sample narrative. The supervisor or his/her designee will look at each manual integration during data review and complete the summary on the data review or QC checklist.

5.1.2 Target: After changing the integration, exit and save in Target Review.

5.1.3 GC/MS Teknivent EnviroQuant/Chemstation: After using Qedit to change the peak click on 'Graphics Report to Printer'

5.1.4 EZChrom: After changing the integration, toggle the "Reanalyze" key and print.

Table 1

Manual Integration Key

M = Manual integration due to missed peak or irregular peak shape.
MS = Manual integration due to split peak.
MR = Manual integration due to retention time shift.
MI = Manual integration of correct isomer.
MT = Manual integration due to peak tailing.

MB = Manual integration due to irregular baseline.

6. REFERENCES

6.1.1 Target Manual, Thru-Put Systems, Inc., Target Compound Analysis Software User's Guide, 1994.

6.1.2 Envirolink User's Manual, TEKNIVENT, 1994

6.1.3 HP Chemstation, Version B.01.00

6.1.4 EZChrom Chromatography Data System, Scientific Software, Rev. 6.6, 1995.

6.1.5 Wisconsin DNR Newsletter, October 1994.

6.1.6 SOP# LTL-1018, Standard Operating Procedure for Review and Approval Practices for Validatable Packages.

6.1.7 SOP # LTL-8005, Standard Operating Procedure for GC Gas/BTEX Data Review.

6.1.8 SOP #LTL-8004, Standard Operating Procedure for GC Volatiles Data Review.

6.1.9 SOP #LTL-8001, Standard Operating Procedure for GC Hydrocarbons Data Review.

6.1.10 SOP #LTL-8301, Standard Operating Procedure for HPLC Aromatics 8310 Data Review.

6.1.11 SOP #LTL-8302, Standard Operating Procedure for HPLC Ordnance 8330 Data Review.

6.1.12 SOP #LTL-8201, Standard Operating Procedure for GC/MS VOA Data Review.

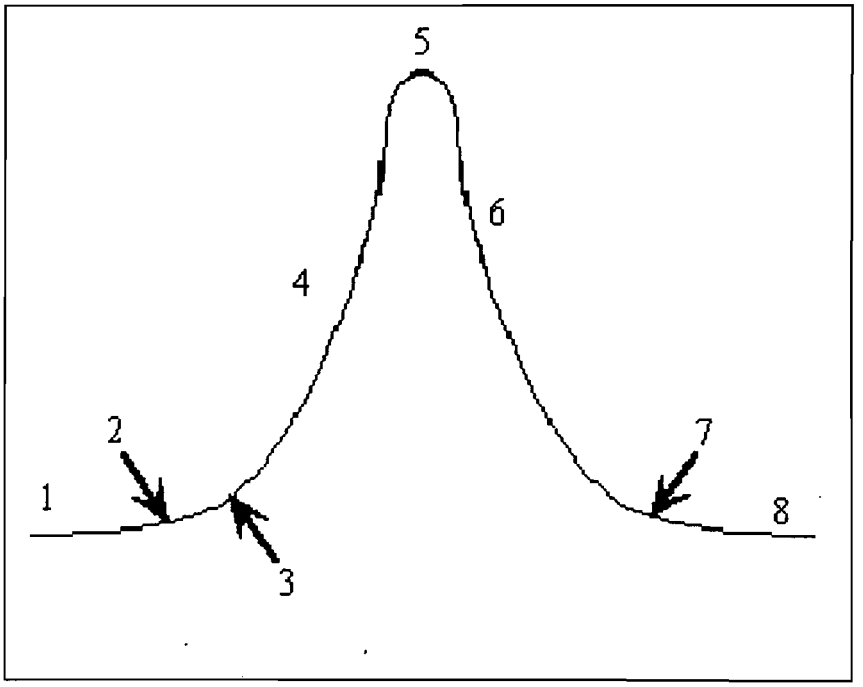
6.1.13 SOP #LTL-8202, Standard Operating Procedure for GC/MS Semivolatile Data Review.

Appendix I

Fig 1.

The sequence for finding a positive peak is:

1. Slope and curvature within limits	track baseline
2. Slope and curvature above limits	perhaps a peak?
3. Slope remains above limit	here's a peak!
4. Curvature becomes negative	front inflection point
5. Slope becomes negative	top of peak
6. Curvature becomes positive	rear inflection point
7. Slope and curvature within limits	approaching end of peak
8. Slope and curvature remain within limits	end peak, track baseline



Steps 3,5, and 8 define Cardinal Points, which are the Start of Peak, Apex, and End of Peak Respectively

Fig 2.

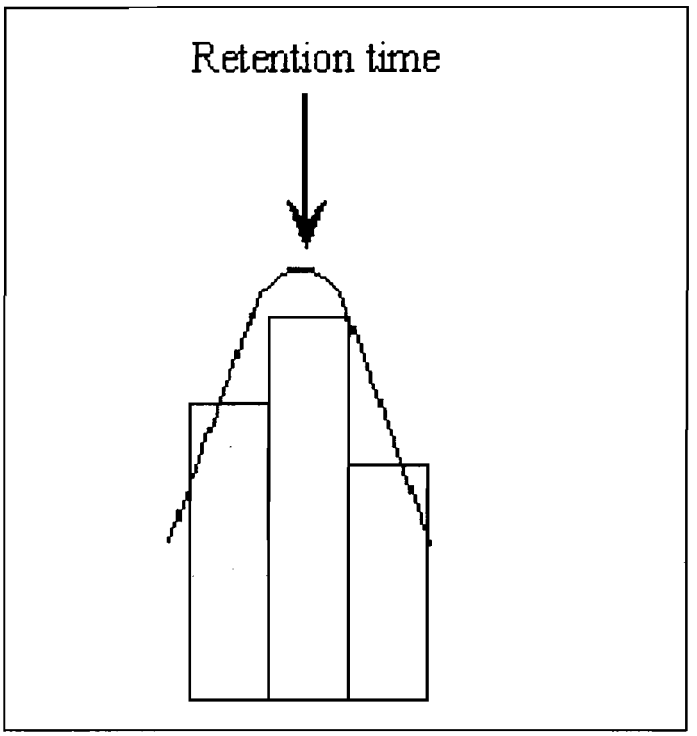
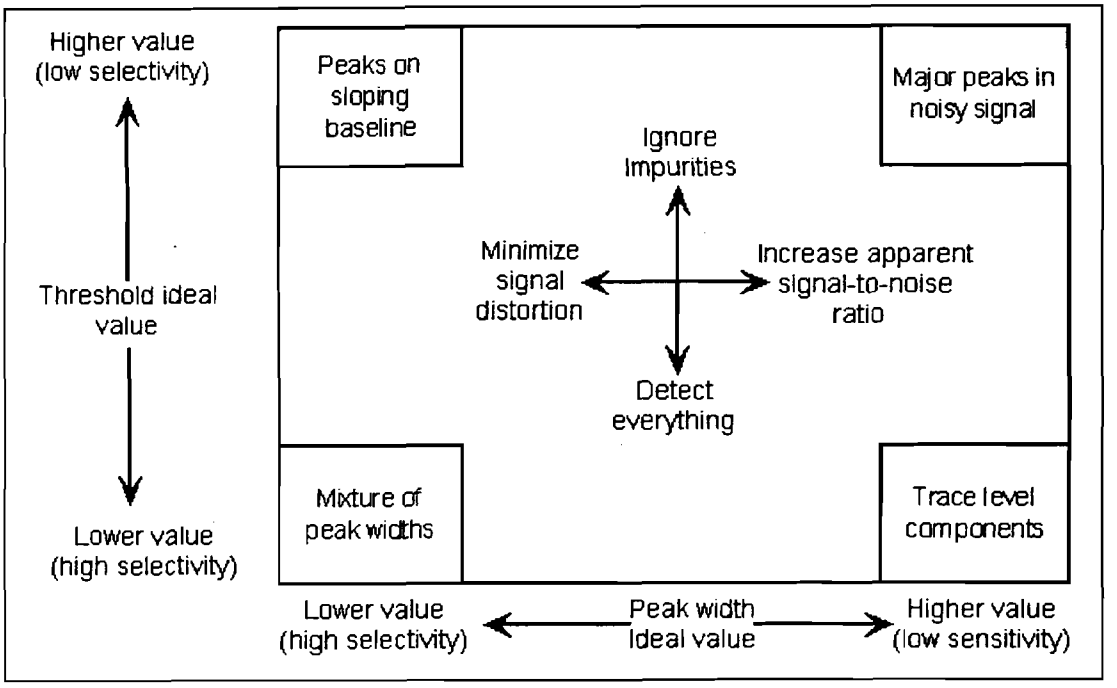
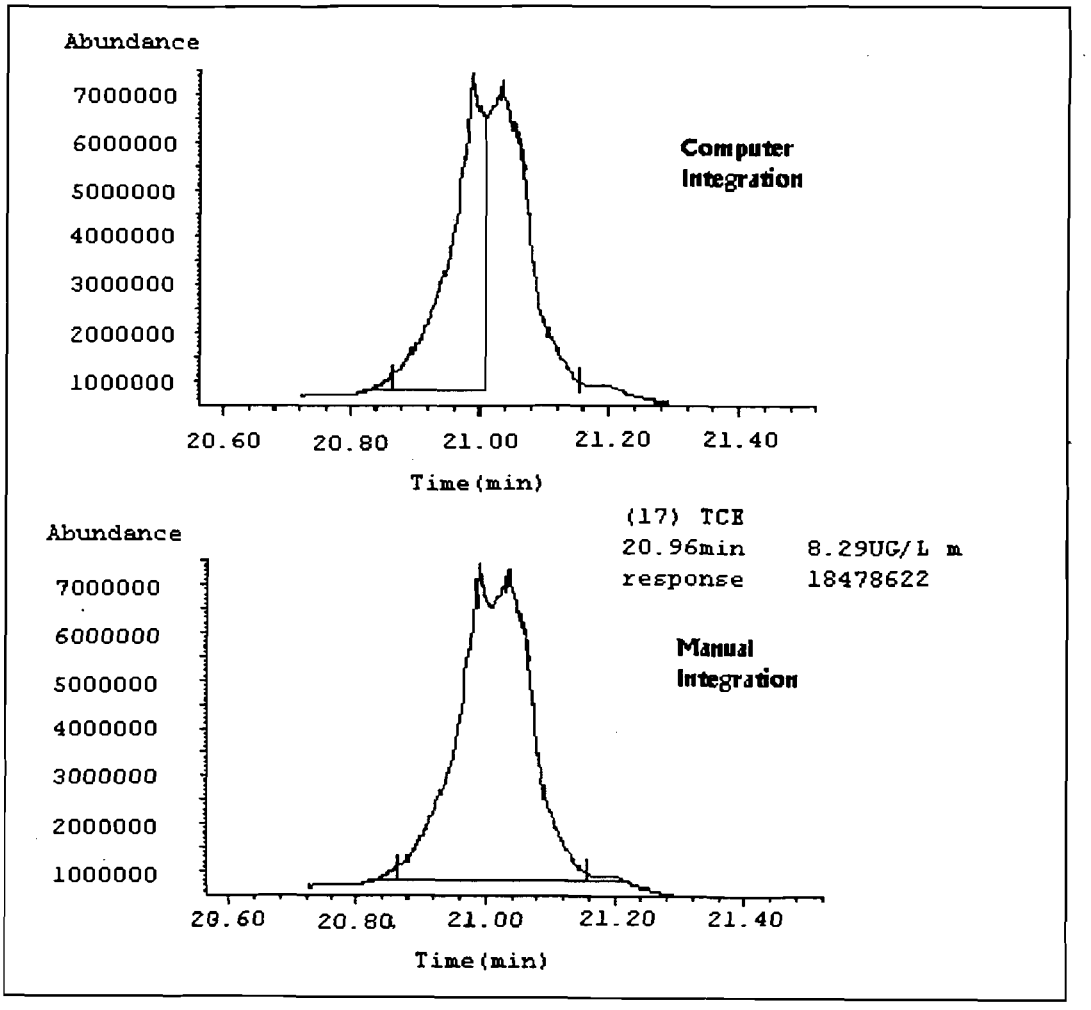


Fig 3.


Higher value
(low selectivity)
Threshold ideal
value
Lower value
(high selectivity)
Peaks on
sloping
baseline
Major peaks in
noisy signal
Ignore
Impurities
Minimize
signal
distortion
Increase apparent
signal-to-noise
ratio
Detect
everything
Mixture of
peak widths
Trace level
components
Lower value
(high selectivity)
Peak width
Ideal value
Higher value
(low sensitivity)

Appendix II

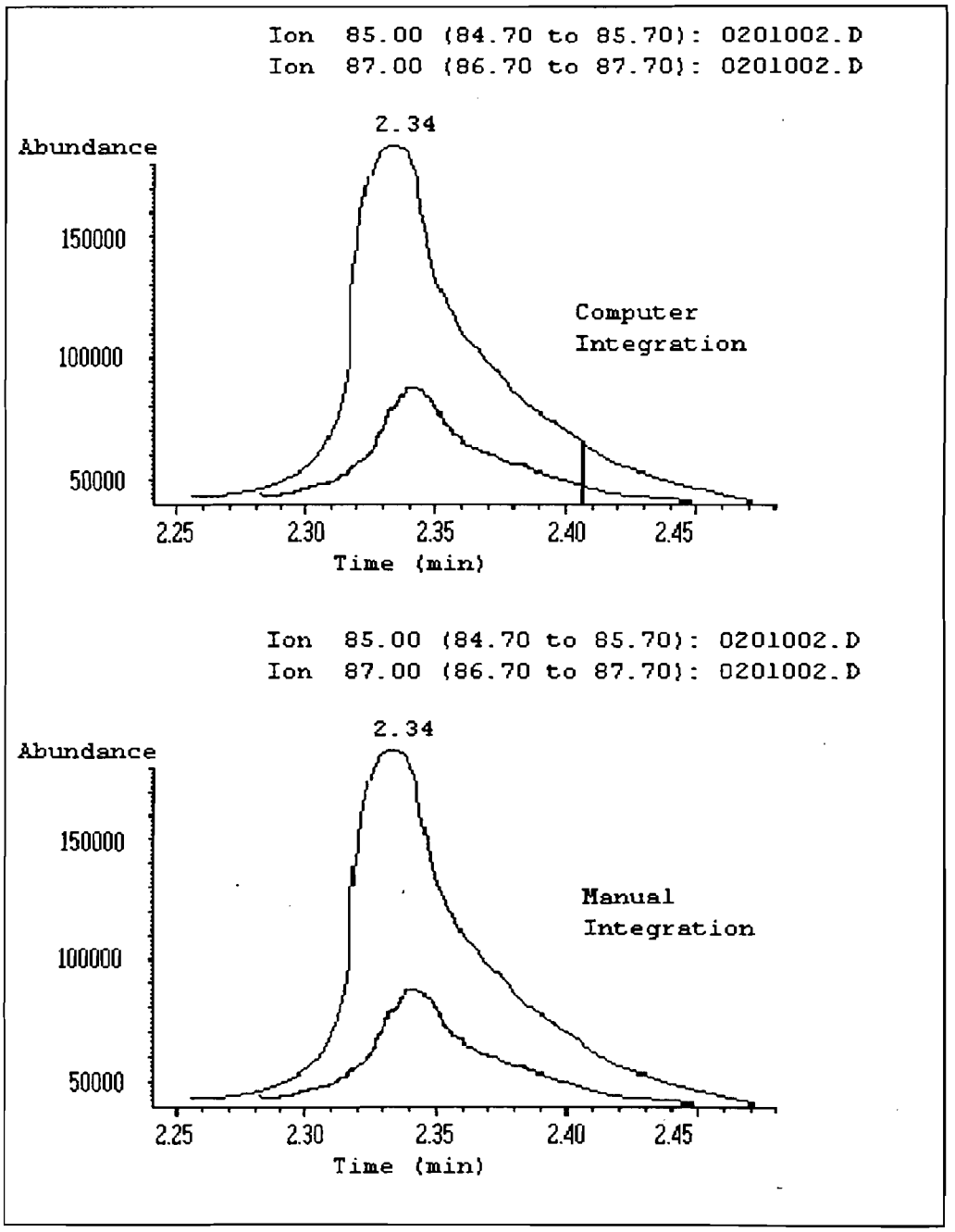
Attachment 1



Allowable Integration

Split Peaks

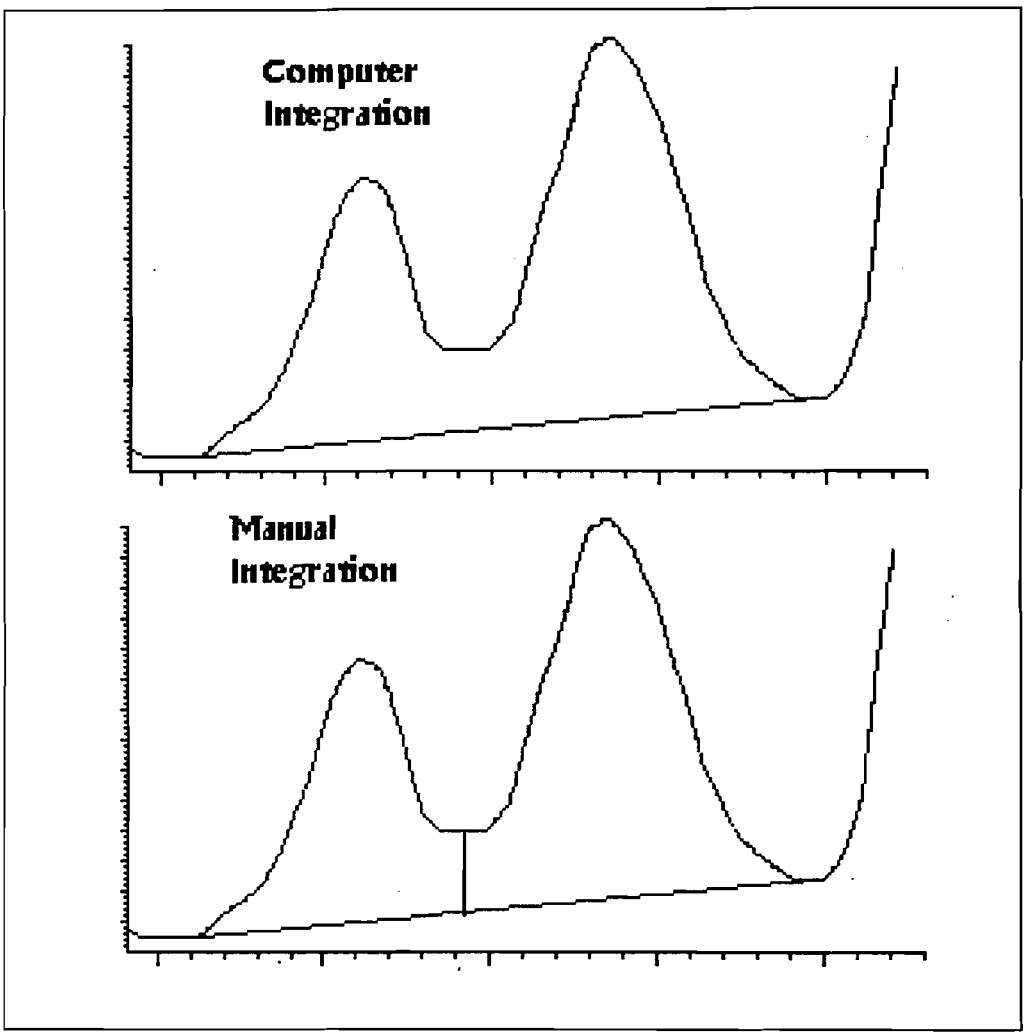
Attachment 2



Allowable Integration

Tailing

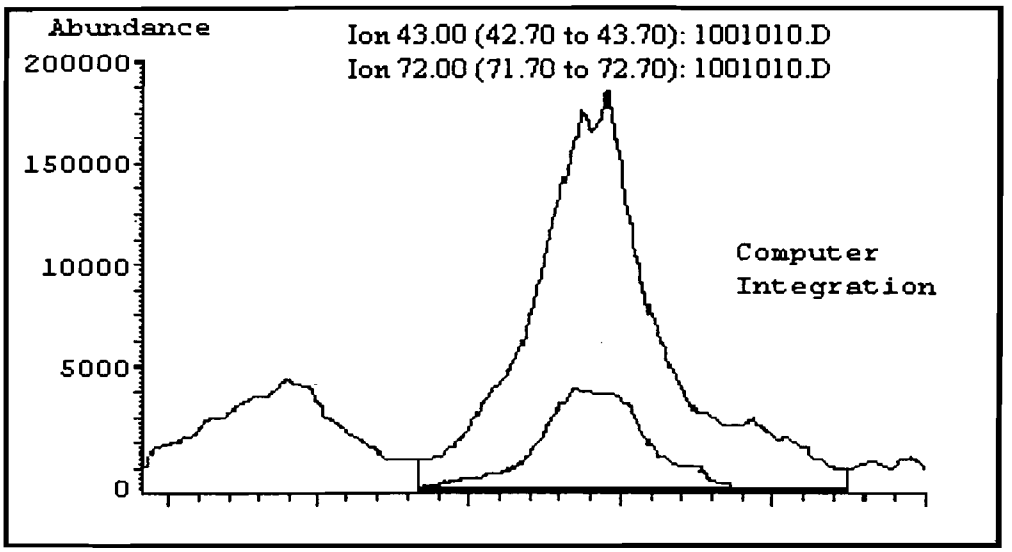
Attachment 3

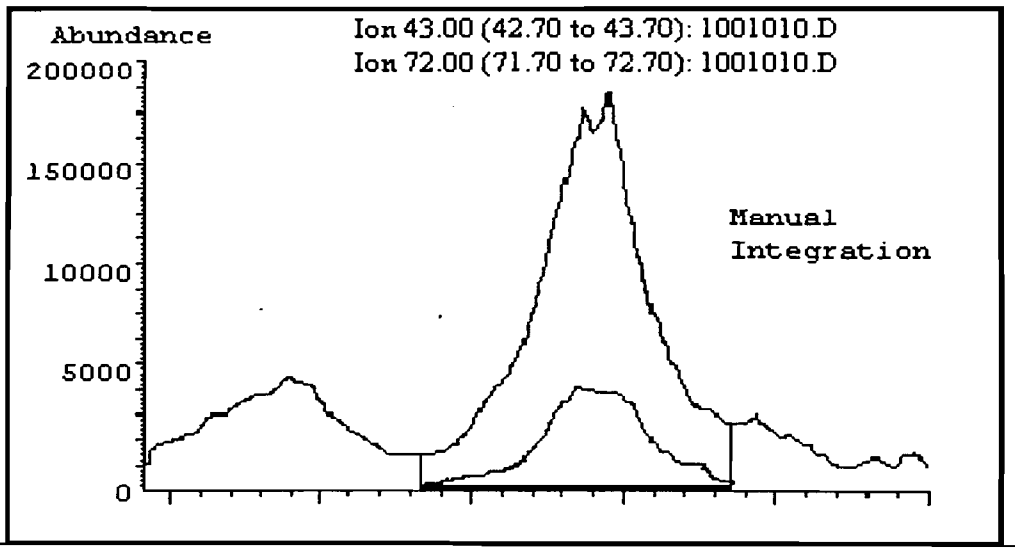


Allowable Integration

Retention Time Shift - Mis-Identification - Merged

Attachment 4

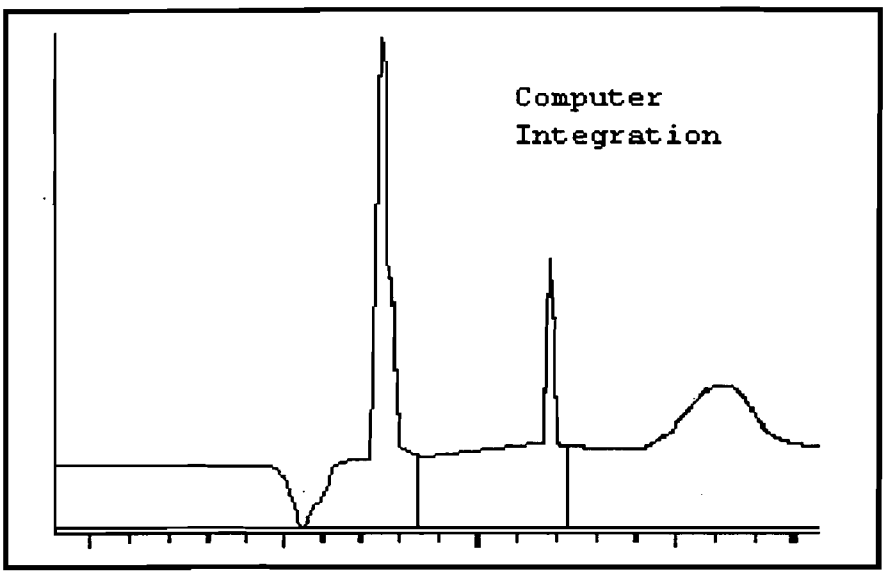


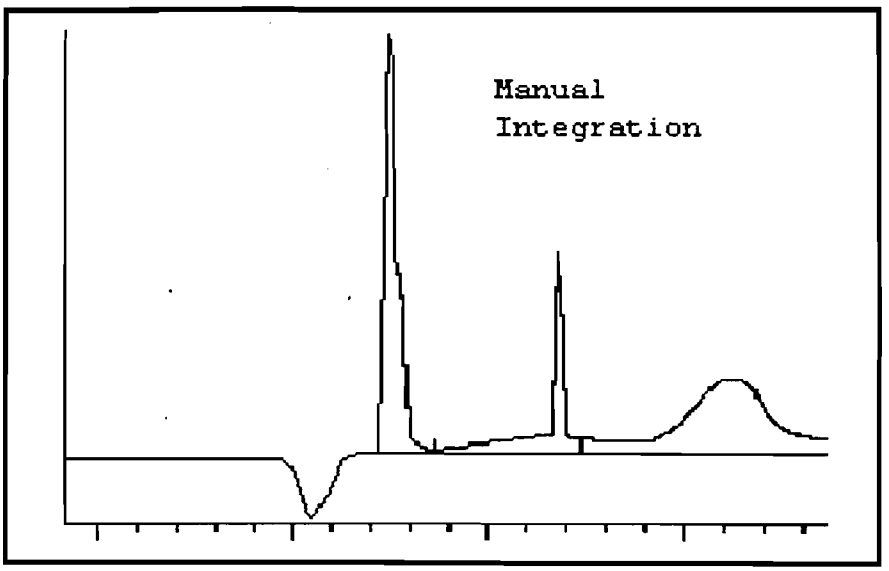


Allowable Integration

Secondary or Qualifier Ions

Attachment 5

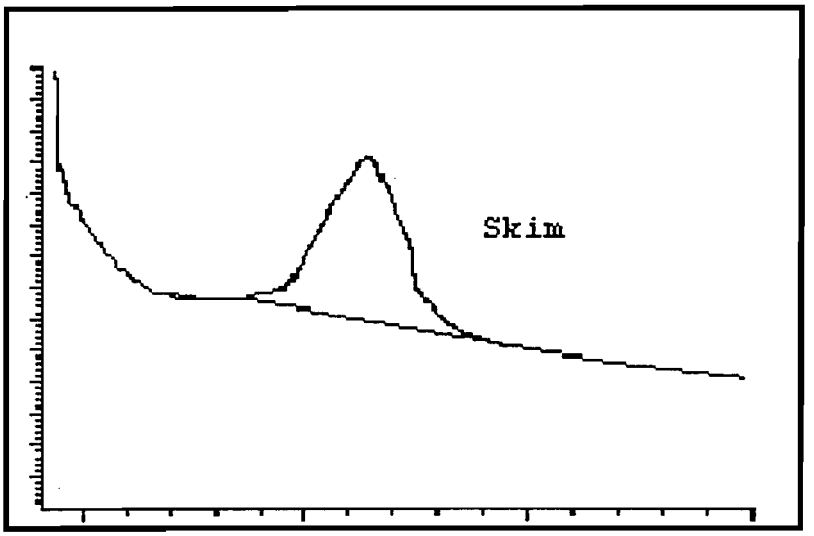


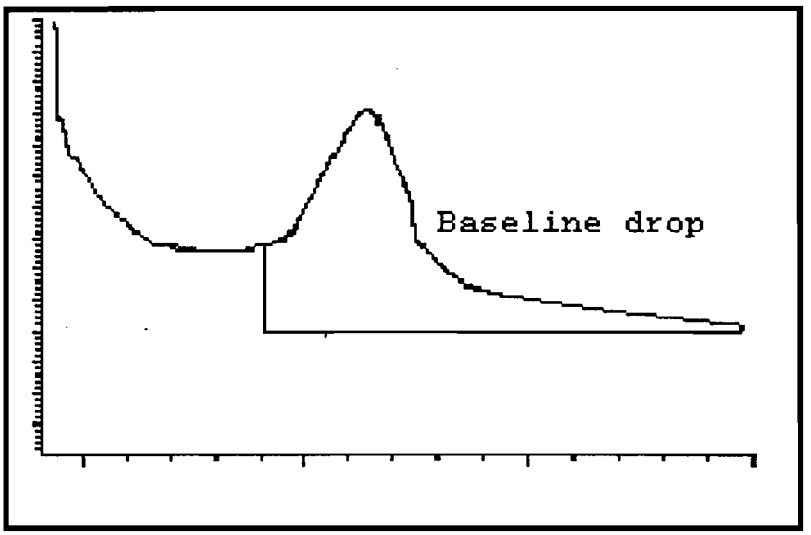


Allowable Integration

Baseline Shift

Attachment 6

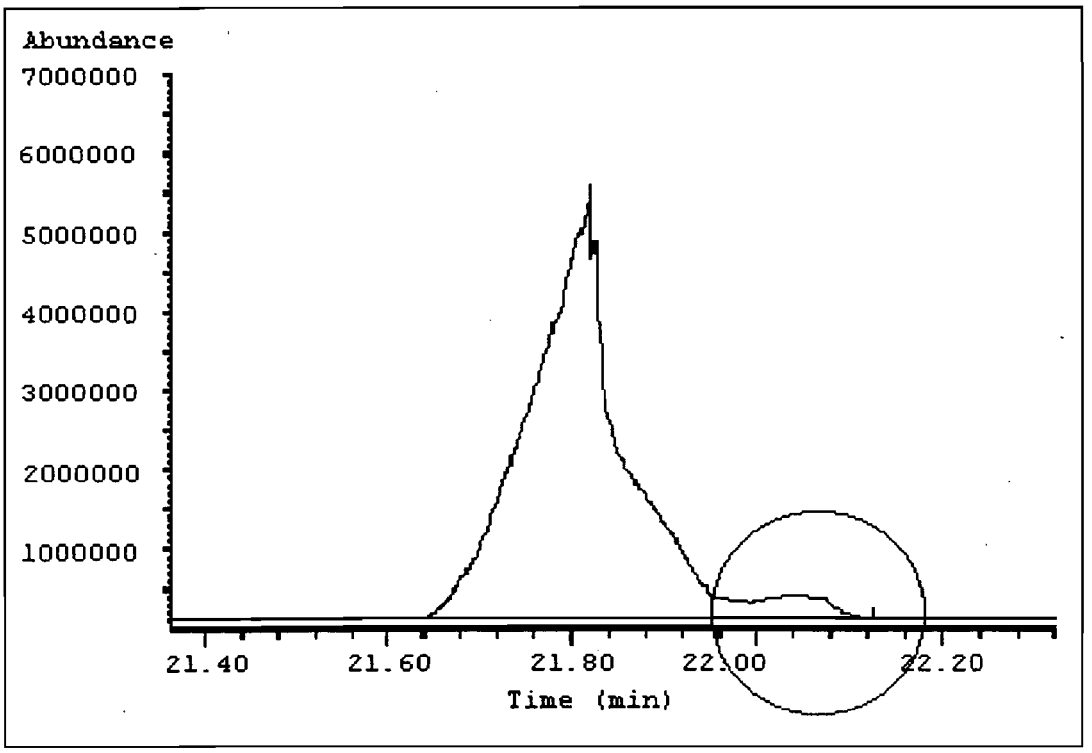




Allowable Integration

Skimming (manual) vs. Baseline Drop (computer)

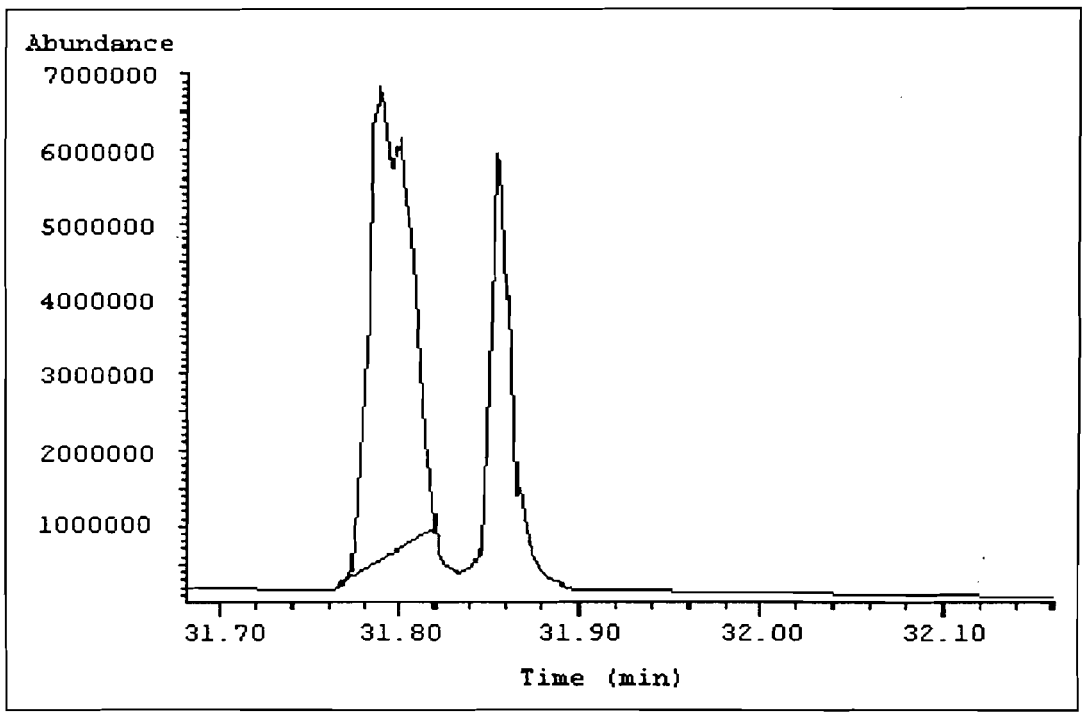
Attachment 7



Improper Integration

Adding Area by Including Other Peaks

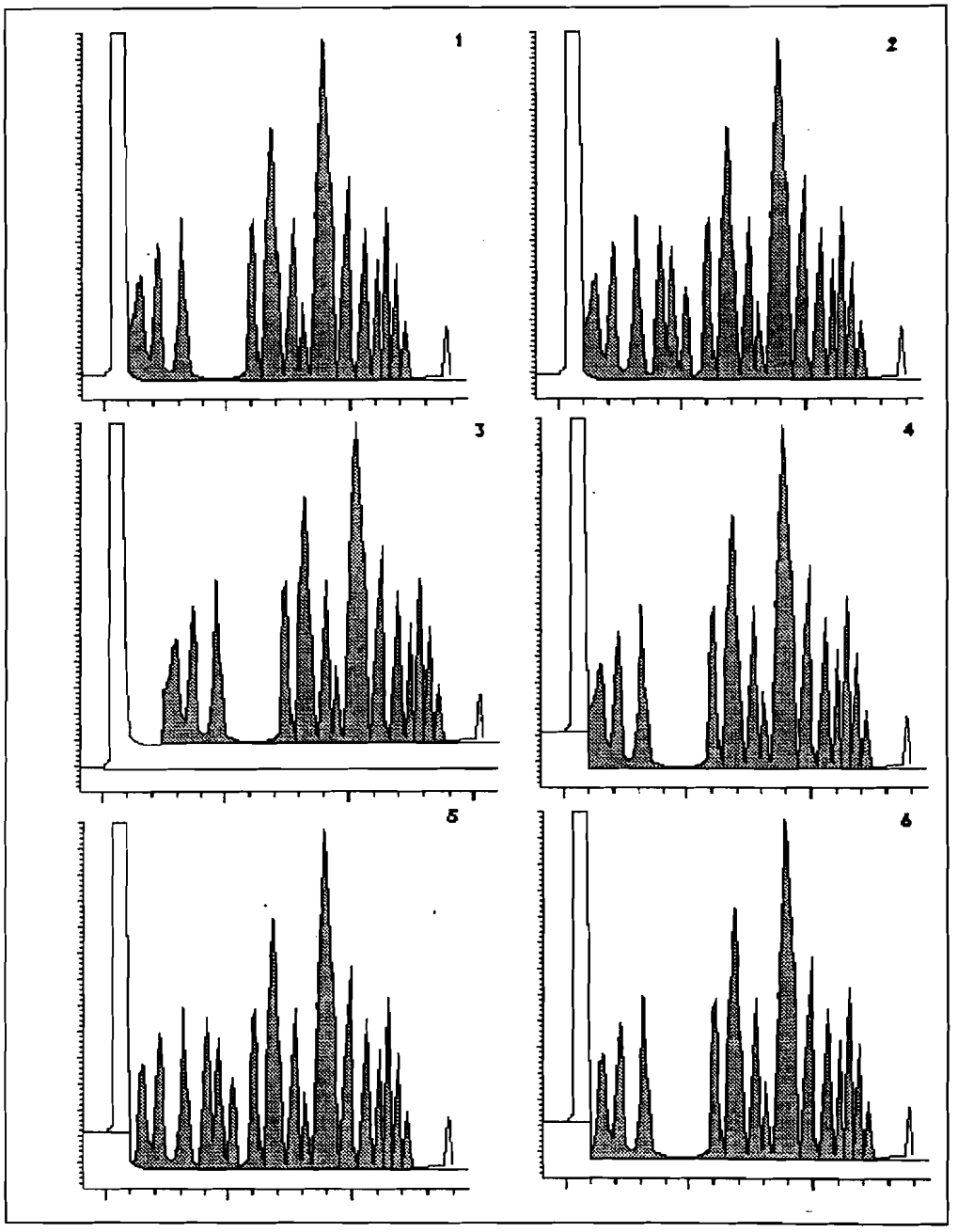
Attachment 8



Improper Integration

Baseline Moved To Decrease Peak Area

Attachment 9



Acceptable Methods of Integration
Grouped Peaks

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

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Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 10-16-01
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: TeTT

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1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD waste, soil samples, aqueous acidic waste, and fluorescent light bulbs. This SOP only covers handling of the waste from the point of collection.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the techniques described. As part of their training for analytical tasks which generate related wastes, each analyst must be trained to properly dispose of the waste or to consolidate it at the appropriate collection point.

1.1.3 This SOP generally does not cover handling of the waste up to the point of disposal.

1.1.4 Appendix III, entitled *"A Quick Primer on Hazardous Waste Management"* contains a brief description of what is expected of individuals in the laboratory in terms of hazardous waste disposal. This is primarily to be used as a reminder and may be handed out independently of the entire SOP. Staff members who handle hazardous waste should also read this SOP.

2. Equipment List

2.1 Equipment

2.1.1 The equipment necessary to properly dispose of laboratory wastes varies with the type of waste. In general, an appropriate container, packing material, and safety equipment (including clothing, eye wear, and respirators) is required.

3. Safety precautions

3.1 Safety Precautions

3.1.1 Solvent wastes may contain materials flammable at room temperature or lower. Caution should be taken to avoid flames and sparks when in the presence of or handling these wastes.

3.1.2 COD and other acidic or alkaline wastes may contain materials which will burn the skin, eyes, and/or mucous membranes if improperly handled. Precautions should be taken to avoid accidental contact.

3.1.3 All wastes may contain materials which can have both known and unknown long-term health effects. COD wastes, for instance, contain high levels of mercury and

chromium salts. Direct contact should be avoided through the use of proper clothing and eye wear, even if no immediate danger is obvious. In the case of volatile solvents and other materials, handling should be done in a well-ventilated area and the exposure to vapors minimized. Where strong fumes are unavoidable, a carbon-filter or other respirator should be worn.

3.1.4 All people who handle waste products or the original reagents should be aware that the laboratory provides safety equipment and has a file containing Material Safety Data Sheets (MSDSs) on all laboratory chemicals in support of OSHA and other safety programs.

4. Operation procedures

4.1 Operations Appropriate to All Collection Areas

4.1.1 Land Disposal Restriction Forms (LDRs), manifests and other paperwork are not extensively discussed in this SOP because the disposal vendor deals with this aspect of the paperwork. It will only be necessary for the person who will be asked by the vendor to sign these forms (usually QA) to check that the information on the forms is accurate and to sign the form.

4.1.2 All waste requiring a Hazardous Waste disposal sticker and manifest will be marked with one of two EPA Hazardous Waste Site numbers. All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608. It is only necessary that these numbers appear on the containers before they are shipped. It is not necessary on drums which have yet to be transported. The labels themselves, however, must be placed on the containers prior to use.

4.1.3 The Hazardous Waste Sticker must be labeled with the proper DOT shipping name, even though the disposal company will usually replace the label before shipping. The proper shipping names are listed below in the applicable sections of this SOP. This item **IS** necessary on all drums as soon as any waste at all is dumped into them.

4.1.4 All collection drums **must** be marked with an appropriately filled out Hazardous Waste sticker (see Appendix A). It is only necessary for Laucks staff to fill in the date that collection was **started** and the contents of the drum in the appropriate space. Hazardous Waste cannot be accumulated for longer than 90 days before it must be disposed. Therefore, do **not** mark the date on the drum until collection is started so as to maximize the allowable time until disposal. This applies to the 55 gal. drums of solvent in the 921 building and the 30 gallon drum in the 940 solvent locker. All other containers are considered satellite collection points and need only be dated when the

containers are **filled**. This sticker will be replaced by the transporter when they arrive to transport the waste to an approved disposal facility. The replacement sticker will contain all of the information required for transport and disposal.

4.1.5 All collection drums containing liquids should have secondary containment. That is, the primary collection drum should sit in a tub or bermed space so that if the container suffered partial or complete failure, there would be sufficient space in the secondary container to hold all of the liquid and not allow it to drain to the sewer.

4.1.6 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive (8) or flammable nature (3) (see Appendix B). Soil drums are designated with a general hazard (9) label.

4.1.7 Once a material has been designated as waste and disposed into the designated drum, that drum must not be stored for longer than 90 days from the date marked on the drum as designated above. This is rarely of concern at Laucks because transport is generally scheduled for most wastes within much less time than the required maximum storage time. Note that Laucks is currently considered a medium quantity generator by the WA Dept. of Ecology and subsequently has 180 days to dispose of full waste containers but we will strive to meet the 90-day clock to insure we are even meeting requirements if we inadvertently exceed the large quantity generator threshold of 2200 lbs./mo.

4.1.8 When collection drums are full or the 90-day limit is approaching, the Quality Assurance (QA) Department must be notified. This department, at the time of this writing, is responsible for contacting the appropriate approved transporter and insuring proper disposal takes place. The preferred lead-time for pickup is 10 working days so QA should actually be contacted 80 days after the first drum is dated. QA does weekly inspections of the laboratory with the express purpose of observing the hazardous waste areas and making safety related observations. However, it should never be assumed that QA or a substitute will automatically take care of a particular accumulation.

4.1.9 All questions or concerns regarding hazardous waste operations should first be directed to QA who will determine the appropriate course of action.

4.1.10 QA or the designated waste manager will also be responsible for insuring that signed manifests are received from the disposal facility within 90-days of shipment. This is easily done by the organization of our hazardous waste files for both of our facilities.

4.2 Mixed Solvent Waste

4.2.1 This waste stream is primarily composed of methylene chloride with some acetone and hexane and potentially small quantities of other solvents or dissolved products. The collection point for all of this waste in the 921 facility (Extractions) are the 55 gallon drums in the solvent locker. In the 940 facility, they are designated 30 gallon drums in the 940 solvent locker

4.2.2 Small, 5 gallon or less containers of other mixed solvent waste may be collected as satellite accumulation units in the inorganics or organics instrument preparation areas but these **must** be transported to the primary drums when full. Likewise, waste solvent bottles are kept in some hoods in the 921 Extractions facility. Satellite accumulation containers must be kept closed when not in use and must be marked with the words "Hazardous Waste" or with other words that identify the contents of the container. This will most conveniently be done by using a blank Hazardous Waste Sticker. Open solvent containers are not allowed except when actually in use.

4.2.3 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of "**Waste Flammable Liquids**".

4.2.4 Although methylene chloride is non-flammable, other components of these waste drums may be highly flammable. Thus, all of the waste solvent containers must be labeled with the flammable hazard (3) sticker.

4.2.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.6 When 3 or more full 55 gallon drums of this waste have been accumulated at 921, or 1 30-gallon drum at 940, or 80 days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.7 At the time of this writing, Laucks uses Safety Kleen as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis.

4.3 Chemical Oxygen Demand (COD) Waste

4.3.1 The primary constituents of this waste are sulfuric acid, water, mercury, silver, and chromium (both tri- and hexavalent). The collection point for this waste is in the inorganics area where CODs are analyzed. These analyses are conducted in small pre-packaged tubes. The reacted tubes are not considered to be waste until they are poured out of the tubes into a collection container.

4.3.2 Collection containers must be labeled with a Hazardous Waste sticker as previously noted. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.3.3 In addition to the hazardous waste sticker, these containers should be labeled with a corrosive (8) sticker as previously noted.

4.3.4 The waste may not be held for more than 90 days from filling of the satellite 5-gallon collection container. Immediately upon filling the container it must be tightly closed, dated, and transported to the acid storage locker. QA must be immediately contacted to arrange for transport and disposal within the allowable timeframe. While being filled, the 5-gallon collection container must be in secondary containment which consists of a washtub in which the collection container sits.

4.3.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.3.6 At the time of this writing, Laucks uses Safety Kleen as the facility of choice for handling this waste stream. This vendor can be changed at the discretion of QA on either a one-time or ongoing basis.

4.4 Soil Sample Disposal

4.4.1 Soil waste drums must be labeled with a hazardous waste label and identified as **"soil contaminated with lead"**. It must also have a general hazard (9) sticker placed right next to the hazardous waste label on the upper half of the drum.

4.4.2 State law allows a laboratory to store samples indefinitely, until they are considered waste and disposed. The laboratory is also allowed to store a 55 gallon drum for at least 90-days (depending on our generator status) after it has been **filled and dated**. The drum must be tightly sealed and an accumulation date written on the hazardous waste label immediately after it has been filled.

4.4.3 Each drum used for soil waste disposal must be clearly marked with an identifying number which will be used to track which drum contained which samples. When samples are signed-out from their storage areas for disposal, the log sheet must be marked with the appropriate assigned drum number. This will enable the laboratory to track which samples were disposed in which drum.

4.4.3.1 The drums should be marked with the year the accumulation is STARTED, location from which they originate, and sequential number. Thus drums for which accumulation began in 1998 from the extractions laboratory would be marked 98-921-01. The -01 being a sequential number that would be incremented with each

additional drum -02, -03, etc. throughout 1998. A drum from the main lab would be designated 98-940-01, etc.

4.4.3.2 When samples are transferred from the storage locations to the drums, the Secure Storage Custody Log must be marked to indicate into which drum they were disposed. This should include any bottle identifiers, if necessary to identify just what was disposed. Thus, it will be necessary for personnel disposing of samples to check the drums to make sure there is enough room for the designated samples. Soil samples will generally have their lids removed and disposed in the regular garbage. If the contents of the jar are easily poured into the drum, this is done and the jar disposed in the waste glass. If not, jar and all will then be disposed in the waste drum. If the lids themselves contain client identifying marks or locations or have significant amounts of adhering material (oil, etc.) which cannot be readily dumped into the drum the lid will also be disposed into the waste drum.

4.4.3.3 When the drums are disposed, it will be necessary for the laboratory representative who signs the manifest to mark the drum identity on the manifest, although this only needs to be on the laboratory copy if the transporter does not want this information to appear on their copy of the record.

4.4.4 QA must be notified 80 days after the drum is full in order to arrange for disposal in a timely manner. This timeframe is not of major concern because there are always Hazardous Waste pickups scheduled within any 90 day time period.

4.4.5 At the time of this writing, Laucks uses Safety Kleen as the vendor of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. This vendor solidifies these soils prior to landfilling in an approved landfill.

4.5 Acidic Waste

4.5.1 All acidic waste must be neutralized before disposal. This includes acid-preserved samples, digests, acid-washings or soaking bathes, etc. unless they contain high levels of heavy metals (which can be disposed with our COD waste if they are not too voluminous, <500 mLs.).

4.5.2 The wastes are poured into a stoppered sink and neutralized with sodium bicarbonate. The resulting neutralized liquid is flushed down the domestic sewer with large quantities of tap water.

4.5.3 Their neutralization and disposal must be documented as it impacts Laucks hazardous waste generator status. This is done in a designated logbook kept near the neutralization sink where the date and final volume disposed are recorded. Note, this

volume should not include the volume of the water used to flush the neutralized solution down the drain.

4.6 Fluorescent Lights

4.6.1 Fluorescent light bulbs are also disposed as hazardous materials. They are in a category termed "universal waste" and are collected in a well marked box similar to that in which they were purchased. When the box is full, it is sent to our hazardous waste vendor or to another recycling firm. This waste stream does not need to be disposed within any particular time frame but should be properly taken care of as soon as the box of bad bulbs is full. Care must be taken to not break the bulbs as they are coated on the inside with hazardous fluorescent material often containing high levels of mercury.

4.7 PCB Oil Waste Disposal

4.7.1 Laucks no longer analyzes many oil samples for PCBs. Thus, this is a very small and infrequent waste stream. However, discussion is presented here in order that there be some documented course of action when it is necessary to dispose of these materials.

4.7.2 All oil samples which are analyzed for PCBs or otherwise known to contain PCBs are treated as PCB oils. No effort is made to distinguish those that actually do contain PCBs.

4.7.3 These oils are accumulated in a 5 gal. drum located in the Extractions laboratory warehouse. This metal drum is stored inside of the lower half of a cut-off plastic 55 gal. drum which fulfills the federal requirements for secondary containment during storage.

4.7.4 When a full drum has been accumulated, Eastern Electric is contacted for pickup and disposal. A signed receipt must be obtained as proof of disposal. Eastern Electric sends a manifest in subsequent mail within 35 days of waste pick-up and must also send a certificate of disposal within 30 days after the actual disposal date.

4.7.5 No annual report to the Department of Ecology is required because the level of PCBs is considered so high as to fall outside of the state's responsibility to monitor. At such levels the federal government regulates the disposal under TSCA. For this reason, it also does not fall within the federal requirement for RCRA governed waste disposal within 90 days. Eastern Electric is responsible for filing appropriate reports. TSCA regulations require that manifests and certificates of disposal be kept on file for a minimum of 3 years.

5. Reports

5.1 Disposal Paperwork

5.1.1 Our current vendor produces all of the required paperwork and insures all of the appropriate container markings (stickers, etc.) are in place prior to shipment. Since Laucks' waste streams are consistent from time to time, our vendors already have the information required to properly fill out the paperwork and Hazardous Waste stickers.

5.1.1.1 The paperwork includes the manifests, land disposal restriction forms and other shipping paperwork. Thus the only requirements of the laboratory are to insure the paperwork is accurate and to sign the appropriate forms.

5.1.2 After the waste has been transported to the disposal or accumulation facility, a signed manifest is returned to the laboratory. This is kept with the permanent record.

5.1.3 All certificates of disposal later provided by the disposal vendor are also associated with any waste shipment and kept with the permanent record.

5.1.4 All records are retained for at least 5 years from the date of shipment of the waste.

5.2 Annual Reporting Requirements

5.2.1 The laboratory must file an annual report with the Washington Department of Ecology (WDOE) for legal and tax purposes. This report is due on March 1 each year. Reports are filed for both the 940 and the 921 facilities (both EPA ID numbers). All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

5.2.1.1 The only exception to this reporting requirement is the reporting of the PCB waste oil which is a federally regulated waste and is thus not reported to the WDOE.

5.2.2 The format of this report is defined by WDOE in books provided to the laboratory several months in advance of the due date. Details of this report are not provided in this SOP.

5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update by September 1 of each year. This report is more loosely defined and the only major requirement is that it be filed. Details of this report are not part of this SOP.

Appendix I

Hazardous Waste Sticker

See directions in this SOP for proper filling out of this sticker.

HAZARDOUS WASTE

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR
PUBLIC SAFETY AUTHORITY, OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

PROPER D.O.T.
SHIPPING NAME_____UN OR NA#_____

GENERATOR INFORMATION:

NAME_____

ADDRESS_____

CITY_____STATE_____ZIP_____

EPA
ID NO._____

EPA
WASTE NO._____

ACCUMULATION
START DATE_____

MANIFEST
DOCUMENT NO._____

HANDLE WITH CARE!

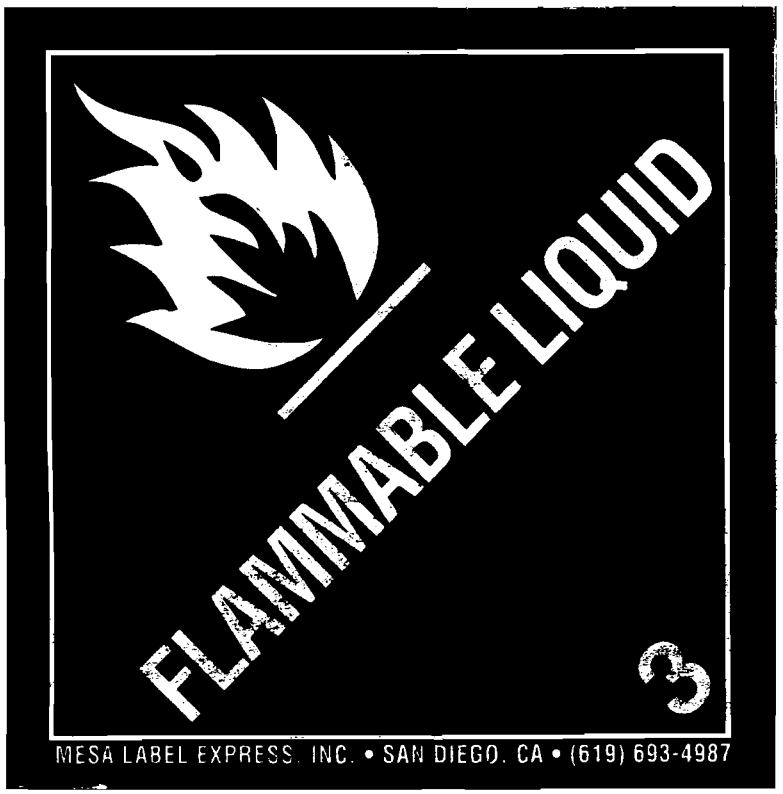
CONTAINS HAZARDOUS OR TOXIC WASTES

STYLE WM-6

Printed by LABELMASTER, Div. of AMERICAN LABELMARK CO., INC., CHICAGO, IL 60646

Appendix II

Example Hazard Class Stickers



8-HML

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Appendix III

A Quick Primer on Hazardous Waste Management

A Quick Primer on Hazardous Waste Management

Laucks is concerned and is indeed obligated by Washington State, to properly manage hazardous waste generated by the laboratory. This document is a quick overview for staff outlining the processes of which individuals should be aware. Everything is outlined in greater detail in Laucks SOP LTL-2001 *Waste segregation and Disposal*.

We currently manage 6 recurring hazardous waste streams at our 940 site and 4 at our 921 site. All individuals should be aware of the need to properly manage hazardous waste and those that need to know more should consult the aforementioned SOP. Any waste not covered in this document or in the SOP or which is not a regularly recurring waste stream must be discussed with QA for proper disposal.

940 Site:

- Solvent Waste – ALL solvent waste is collected in the 30 gallon drum in the solvent locker clearly labeled "Hazardous Waste". Solvent is collected in satellite containers located under the organic standards hood and in the waste solvent containers from the HPLC. Waste solvent should not be accumulated anywhere else without permission from QA. All other areas where solvent might be used should be immediately transferring the solvent to the 30 gallon container. Solvent is not to be dumped into any drum until it has been properly labeled.

- COD Waste – This is waste from the analysis for Chemical Oxygen Demand and is not only acidic but contains high concentrations of several metals, including Cr^{+6} and Hg. It is collected in a clearly labeled 5 gallon carboy underneath the sink where CODs are analyzed. There should be no other collection point. When the container is filled to 2 inches from the top, it should be tightly capped, the current date marked under the "accumulation start date" on the Hazardous Waste label, and the carboy transferred to the acid storage locker. A new 5 gallon container should be started and QA should be contacted for the required labeling. Waste is not to be dumped into any carboy until it has been properly labeled.

- Soil – Waste soil samples are assumed to contain hazardous metals, specifically lead. Unless a soil is known to have been an agricultural soil or is known not to contain hazardous substances, it is treated as though it is hazardous. The soil samples are dumped into a 55 gallon drum in the 940 warehouse. When the drum is full, it is sealed, the current date marked under the "accumulation start date" on the Hazardous Waste label, and QA notified in order to arrange transport to a disposal facility. A new drum will be labeled by QA. Soil is not to be dumped into any drum until it has been properly labeled.

- Extract Vials – Extract vials are collected in the covered, labeled 5 gallon bucket in the organic standards hood. When the bucket is full to the point that it can just be closed and sealed, it should be. The current date should then be marked on the Hazardous Waste label and QA contacted to arrange for disposal. A new 5 gallon container should be started and QA should be contacted for the required labeling. Waste is not to be dumped into any container until it has been properly labeled.

- Acidic Waste – Acidic waste containing significant amounts of metals should be disposed separately. QA should be consulted to make appropriate arrangements. Waste which is only acidic MUST first be neutralized with bicarbonate and then can be poured down the drain into the domestic sewer. This waste, however, must be recorded in a logbook kept for that purpose. The SOP should be consulted for further information.

- Fluorescent Lights – Fluorescent lights are considered "Universal Waste" and must never be disposed in the garbage. They must be collected and shipped to a recycling or hazardous waste disposal facility. These are collected in the boxes in which the replacement bulbs come, located in the 940 warehouse.

921 Site:

- Solvent Waste – ALL solvent waste is collected in the 55 gallon drum in the solvent locker clearly labeled "Hazardous Waste". Solvent may be collected in satellite containers located under some hoods but should NOT be allowed to sit uncovered and any such containers must be dumped into the 55 gallon container each night before the last analyst leaves. Solvent is not to be dumped into any drum until it has been properly labeled. Labeling instructions are in the SOP and labels are located in a drawer underneath the PCB hood.

- Soil – Waste soil samples are assumed to contain hazardous metals, specifically lead. Unless a soil is known to have been an agricultural soil or is known not to contain hazardous substances, it is treated as though it is hazardous. The soil samples are dumped into a 55 gallon drum in the 921 warehouse. When the drum is full, it is sealed, the current date marked under the "accumulation start date" on the Hazardous Waste label, and QA notified in order to arrange transport to a disposal facility. A new drum will be labeled by QA. Soil is not to be dumped into any drum until it has been properly labeled.

- Fluorescent Lights – Fluorescent lights are considered "Universal Waste" and must never be disposed in the garbage. They must be collected and shipped to a recycling or hazardous waste disposal facility. These are collected in the boxes in which the replacement bulbs come, located in the 940 warehouse.

- PCB Oil Waste – This waste is collected in a 5 gallon pail located in a blue plastic half-drum in the 921 warehouse. Only oil known or suspected to contain PCBs should be placed in this pail. Since we rarely get these oils anymore, the use of this container should be very infrequent. When it is full, QA is contacted to arrange for disposal and a new pail.

Extraction Method for Polynuclear Aromatics in Water
(8270C by 3520C)

Method # LTL-3001

January 26, 2001

Revision Number: #4

Written by: _____ Date: 1.26.01

Reviewed by: _____ Date: 4-30-01

Approved by: _____ Date: 04-30-01

Controlled Document

No. 20 Assigned to: TETR

1. **PURPOSE** - In this method, polynuclear aromatics are extracted at pH <2 with methylene chloride using a continuous liquid-liquid extractor. Extracts are dehydrated and concentrated in a Kuderna-Danish (K-D) apparatus for analysis by GC/MS. This method also includes low concentration (SIM) method.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2. All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1 1:1 Sulfuric Acid - measure equal volumes of concentrated sulfuric acid and DIW. Note: Observe proper acid preparation techniques.

3.3.2. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400° C.

3.3.3. Alumina, Neutral, Brockman Activity I - activate 60-325 mesh alumina by heating for 16 hours at 130° C.

3.3.4. Surrogate solution prepared in methanol:

1-Fluoronaphthalene	250 ug/ml
Fluorene-d10	250 ug/ml
Pyrene-d10	250 ug/ml

Low Concentration (SIM) surrogate solution prepared in methanol:

1-Fluoronaphthalene	12.5 ug/ml
Fluorene-d10	12.5 ug/ml
Pyrene-d10	12.5 ug/ml

3.3.5. Matrix spiking solution and Low Concentration (SIM) matrix spiking solution prepared in methanol:

Acenaphthene	50 ug/ml
Acenaphthylene	50 ug/ml
Anthracene	50 ug/ml

Benzo(a)anthracene	50 ug/ml
Benzo(a)pyrene	50 ug/ml
Benzo(b)fluoranthene	50 ug/ml
Benzo(ghi)perylene	50 ug/ml
Benzo(k)fluoranthene	50 ug/ml
Chrysene	50 ug/ml
Dibenzo(a,h)anthracene	50 ug/ml
Fluoranthene	50 ug/ml
Fluorene	50 ug/ml
Indeno(1,2,3-cd)pyrene	50 ug/ml
Naphthalene	50 ug/ml
Phenanthrene	50 ug/ml
Pyrene	50 ug/ml
2-Methylnaphthalene	50 ug/ml

4. **EQUIPMENT**

4.1. Continuous liquid-liquid extractor with 500 ml round, flat-bottom boiling flask

4.2. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.3. Teflon thistle tube

4.4. Standard laboratory glassware to include:

4.4.1. K-D apparatus: 500 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.4.2. Monstr-pette disposable pipettes.

4.5. All glassware to be rinsed as follows, prior to use:

4.5.1. Technical grade acetone (if the glassware is wet).

4.5.2. Triple rinsed with methylene chloride.

4.6. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction:

6.1.1. Mix sample thoroughly in its original container and place labeling tape at sample level. Mark meniscus of sample on the tape.

6.1.2. Transfer entire contents of container into a liquid-liquid extractor. Rinse container with methylene chloride and add to extractor. Fill sample container to the mark on tape with water. Measure volume of sample used in a calibrated 1000 ml cylinder.

6.1.3. Prepare a blank and a blank spike with 1000 ml DIW.

6.1.4. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.5. Pipet 200 ul surrogate solution into each extractor.
(Low Conc: 100 ul of low conc. surrogate solution.)

6.1.6. Pipet 500 ul matrix spiking solution into each QC extractor.
(Low Conc: 25 ul of matrix spiking solution.)

6.1.7. Adjust the sample to pH <2 with 1:1 sulfuric acid.

6.1.8. Add 300 ml methylene chloride and boiling chips to a round, flat-bottom boiling flask.

6.1.9. Turn on the water recirculator and extract samples for 18 hours.

6.1.10. Allow the assembly to cool and collect all solvent in the boiling flask.

6.2. Solvent Dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from step 6.1.10. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent Evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 8 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml, remove the apparatus from the water bath and cool to room temperature.

6.3.4. Rinse joint and remove the snyder column. Allow rinse solvent to drain into ampule.

6.3.5. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of methylene chloride.

6.3.6. Reduce the extract volume to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of ampule several times during blowdown.

6.3.7. Transfer extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methylene chloride.

Low Conc: Final volume adjusted to 0.5 ml. in methylene chloride.

6.3.8. Proceed to 6.4.10.

NOTE: If there is significant color in the extract, an alumina cleanup (Method 3610B) is indicated.

6.4. Alumina Cleanup

6.4.1. Prepare a Monstr-pette column by plugging with a small amount of glass wool.

6.4.2. Pack column with a minimum of 3 gm. of activated alumina. Top with 1/2 cm of sodium sulfate.

6.4.3. Pre-elute column with 10 ml methylene chloride.

6.4.4. Add entire extract directly to top of the sodium sulfate and allow to elute onto the column.

6.4.5. Rinse the ampule twice with a small amount of methylene chloride and add to top of column.

6.4.6. Attach a Teflon thistle tube to the column and add a minimum of 10 ml methylene chloride.

6.4.7. Elute into a 16 x 125 mm culture tube.

6.4.8. Reduce the extract volume to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of the culture tube several times during blowdown.

6.4.9. Transfer extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methylene chloride.

Low Conc: Final volume adjusted to 0.5 ml. in methylene chloride.

6.4.10. Label the extract and deliver to 940.

6.4.11. Complete all paperwork and bench sheet. Bench sheet should include cleanup method if indicated, alumina lot #, date and time of transfer to 940 and extract location. Clip T-card on the folder and place in GC/MS room extraction folder box. File folder color is dark blue and blank name ____MPNWL_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846.

3520C (1996), 8270C (1996), 3610B (1996)

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 01013367	MSPNA	1-25-01	3520C	LS/AGH	BD 1-27-01	MSPNA
MATRIX: H2O						

Sample #	wt./vol.	Final Vol				Ext #	SDG #	Bkl #
01013367-1	1020	1.0 ml				3	T1008	62
2	1010					4		73
3	980					5		79
4	1040					6		84
4MS	1010					7		85
4MSD	1000					8		86
5	1010					9		90
S012501MPNWLS	1000					10		
B012501MPNWLS	1	1				11		

BD 1-25-01

COMMENTS:

Ext & Final Sol: MeCl2 - Bxs # BZ743
- GN <2 - Liq/Liq on 1330; off 0730 1-26-01

SURROGATES:

200 ul of: Ex 8-46.1

_____ ul of: _____

SPIKES:

500 ul of: Ex 8-38.3

_____ ul of: _____

STANDARDS:

_____ ul of: _____

_____ ul of: _____

_____ ul of: _____

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
MSPNA	Time out: 0830	Time in: Empty	Time out: 1320	Time in: —	Tray # 1542 @4 -C12	Time out:	Time in:
	Date: 1-25-01	Initials: LS	Date: 1-27-01	Initials: AGH		Date:	Initials:
	Time out:	Time in:	Time out: —	Time in:		Time out:	Time in:
	Date:	Initials:	Date:	Initials:		Date:	Initials:

Extraction Method for Chlorinated Herbicides in Soil (8151A by Modified 3550C)

Method # LTL-3011

October 16, 2001

Revision Number: #2

Written by: [illegible] Date: 10-16-01

Reviewed by: Harry Romberg Date: 10-16-01

Approved by: Kathy Freps Date: 10-16-01

Controlled Document

No. 20 Assigned to: Tetr

1. **PURPOSE** - In this method, samples are first subjected to an alkaline hydrolysis. The sample is extracted while alkaline to remove extraneous materials. It is then acidified, forming the less polar species, which are extracted into ethyl ether. The extracts are derivatized with diazomethane and the methyl esters are isolated for analysis by GC/ECD.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples, by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **MATERIALS**

3.1. All reagents are to be AR grade or better.

3.2. All solvents are to be distilled in glass unless otherwise noted.

3.3 The following special reagents should be prepared:

3.3.1. 37% Potassium Hydroxide - prepared by dissolving 370 grams potassium hydoxide pellets and diluting to one liter with DIW.

3.3.2. Concentrated Sulfuric Acid

3.3.3. 10% Hydrochloric Acid - measure 100 ml concentrated hydrochloric acid and dilute to 1000 ml with DIW. Note: Observe proper acid preparation techniques.

3.3.4. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.5. Acidified Sodium Sulfate - Weigh the muffled anhydrous sodium sulfate in 100 gram increments into a suction flask. Cover the sodium sulfate with ethyl ether containing 0.1 ml concentrated sulfuric acid for each 100 g increment. Swirl the slurry for about two minutes then stopper the flask and apply an aspirator vacuum until the ether is completely removed . Place on a steam bath in a hood to assure that all ether is evaporated. Store in a closed bottle.

3.3.6. 10 M Potassium Hydroxide - prepared by dissolving 56.1 grams potassium hydoxide pellets and diluting to 100 ml with DIW.

3.3.7. Diethylene Glycol Monoethyl Ether (Carbitol) – (Sigma – catalog # D-1265)

3.3.8. Diazald® (Aldrich - catalog # D2,800-0).

3.3.9. Activated Silicic Acid - activate 100 mesh silicic acid powder by heating for 16 hours at 130°C.

3.3.10. Surrogate solution prepared in methanol:

2,4 - Dichlorophenyl Acetic Acid	5.0 ug/ml

3.3.11. Matrix Spiking solution prepared in methanol:

2,4,5-TP	0.8 ug/ml
2,4,5-T	0.8 ug/ml
Dicamba	0.8 ug/ml
Pentachlorophenol	4.0 ug/ml
Dinoseb	4.0 ug/ml
Picloram	8.0 ug/ml
2,4-DB	8.0 ug/ml
2,4-D	8.0 ug/ml
Dichloroprop	8.0 ug/ml
Dalapon	20.0 ug/ml
MCPP	800.0 ug/ml
MCPA	800.0 ug/ml

4. EQUIPMENT

4.1. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.2. Standard laboratory glassware to include:

4.2.1. 40 ml Teflon culture tubes

4.2.2. 16 x 100 mm culture tubes

4.3. All glassware to be used shall be rinsed in the following manner:

4.3.1. 10% hydrochloric acid

4.3.1. Reagent grade acetone

4.4. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blank, blank spike, matrix spike and matrix spike duplicate (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction:

6.1.1. Mix the sample thoroughly in its original container if there is space available, otherwise mix in solvent rinsed aluminum tray.

6.1.2. Weigh 15.0 grams of soil (wet weight) into an Teflon culture tube. Cover sample with DIW.

6.1.3. Prepare two additional aliquots of a sample if Quality Control is required.

6.1.4. Prepare a blank and a blank spike with 10 ml. DIW.

6.1.5. Pipet 100 ul surrogate solution into each tube.

6.1.6. Pipet 100 ul matrix spiking solution into each QC tube.

6.1.7. Pipet 1.0 ml 37% potassium hydoxide into each culture tube, mix and loosely cap with teflon screw cap.

6.1.8. Immerse tubes in a 60-65°C water bath for a minimum of 90 minutes.

6.1.9. Remove from bath and cool to room temperature.

6.1.10. Add 6-7 mls ethyl ether to each culture tube, cap tightly and shake for two minutes. Sonicate in water bath for 5-10 minutes. If emulsions occur, centrifuge tubes.

6.1.11. Draw off and discard ether layer.

6.1.12. Repeat from step 6.1.10. two additional times using 3 ml ethyl ether.

6.1.13. Adjust pH to <2 by slowly adding 500µl concentrated sulfuric acid to each tube. Mix and check pH.

6.1.14. Repeat step 6.1.10 three times using 3 ml ethyl ether and save ether into a 16 x 100 culture tube.

6.2. Dehydration of the extract:

6.2.1. Adjust volume to 5-6 mls in the nitrogen blowdown.

6.2.2. Add 3 gm of acidified sodium sulfate to each tube, cap and shake.

6.2.3. Tap the tubes to settle the sodium sulfate and let stand at least 60 minutes and may stand overnight.

6.2.4. Transfer ether extract to 16 x 100 culture tube.

6.2.5. Add 1-2 mls ether to acidified sodium sulfate, cap and shake to break up sodium sulfate.

6.2.6. Transfer rinse to tube containing the ether extract.

6.2.7. Repeat from step 6.2.5. two additional times.

6.2.8. Gently reduce the volume to approx. 1 ml in the nitrogen blowdown.

6.2.9. Add 0.5 ml iso-octane and bring to 2.0 ml with ethyl ether.

6.3. Derivatization:

6.3.1. To tube A (the first tube in the train), add 1.0 ml methanol, and bring to 8.0 ml with ethyl ether.

6.3.2. To tube B add:

6.3.2.1. 2.0 ml 10 M potassium hydoxide (to the first marked line)

6.3.2.2. 1.4 ml Carbitol (diethylene glycol monoethyl ether) to the second marked line.

6.3.3. Place 10 ul micro-pipet in each sample tube from step 6.2.8.

6.3.4. Connect nitrogen line to the apparatus and regulate so that tube A bubbles actively but not violently.

6.3.5. Add 2 scoops (approximately 0.1-0.2 grams) of Diazald and 1.4 ml ethyl ether to tube B. Stopper, connecting tube A to tube B.

6.3.6. Bubble the generated diazomethane into the tube for 90 seconds. (Extract will turn bright yellow.)

6.3.7. Let extract stand for 15 minutes.

6.3.8. Add 0.1 - 0.2 gm. activated silicic acid to each culture tube. Allow to stand until evolution of nitrogen gas has stopped. This may take several hours and may be stored overnight.

6.3.9. Adjust the final volume to 5.0 ml (as compared to a measured volume) with hexane and centrifuge.

6.3.10. Label the extract and deliver to 940.

6.6.11. Complete all the necessary paperwork and bench sheet. Bench sheet to include date derivatized, time and date of transfer to 940 and extract location. Clip the T-card on the folder and place in the GC room extraction folder box. The file folder color will be yellow, and the blank name will be ____GHB.SL_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846.

3550C (2001), 8151A (1996)

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 0106262	Vert	6-18-01	mod 3550C	YL	SD 6-19-01	8151A
MATRIX: Soil						

Sample #	wt./vol.	% Vol					SDG #	Bottle #
0106262-1	15.0g	5.0ml			—		T5904	32
2								33
3								34
4								35 & 36
4MS								
4MSD								
5								37
6								38
S061801GWBS4P								
B061801GWBS4P								

6-18-01

YL

COMMENTS:

Ext Sol: Ethyl Ether - BGS # CB192
Fumed Sol: Hexane - EM# 40163
Derivatized - 6-20-01

SURROGATES:

100 ul of: Ext 8-51-5
ul of:

SPIKES:

100 ul of: Ext 8-47-1
ul of:

STANDARDS:

ul of:
ul of:
ul of:

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
	Time out	Time in	Time out	Time in	Tray #	Time out	Time in
Vert	0900	1230	1450	—	6-19-01		
	Date	Initials	Date	Initials	D2-E1	Date	Initials
	6-18-01	SD	6-20-01	YL			
	Time out	Time in	Time out	Time in		Time out	Time in
			—	1450			
	Date	Initials	Date	Initials		Date	Initials
			6-20-01	SMS			

Extraction Method for Ordnance Compounds in Water (8330A)

Method # LTL-3077

May 4, 2001

Revision Number: #5

Written by: B. O Gill Date: 5-4-01

Reviewed by: Harry Pinkney Date: 5-7-01

Approved by: Kathy Grapes Date: 5-8-01

Controlled Document

No. 20 Assigned to: TETR

1. **PURPOSE** - In this method the ordnance compounds are extracted from neutral water with acetonitrile. The extract is salted-out and analyzed by HPLC.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2. All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. Sodium Chloride - prepared by muffling AR grade sodium chloride for four hours at 400°C.

3.3.2. Salt Water - prepared by weighing 325 grams muffled sodium chloride, dissolving in DIW and bringing to 1000 ml.

3.3.3. Surrogate solution prepared in methanol:

1,2-Dinitrobenzene	400 ug/ml

3.3.4. Matrix spiking (MS) solution prepared in methanol:

1,3-Dinitrobenzene	80 ug/ml
2,4-Dinitrotoluene	80 ug/ml
2,6-Dinitrotoluene	80 ug/ml
HMX	80 ug/ml
RDX	80 ug/ml
Nitrobenzene	80 ug/ml
2-Nitrotoluene	80 ug/ml
3-Nitrotoluene	80 ug/ml
4-Nitrotoluene	80 ug/ml
Tetryl	80 ug/ml
TNT	80 ug/ml
1,3,5-Trinitrobenzene	80 ug/ml
2-amino-4,6-dinitrotoluene	80 ug/ml
4-amino-2,6-dinitrotoluene	80 ug/ml

3.3.5. Special Matrix spiking solution for PETN/NG in methanol:

PETN	50 ug/ml
Nitroglycerin	100 ug/ml

3.3.6. Special Matrix spiking solution for 6 Attenuation compounds in methanol:

2,4-DA-6-NT	80 ug/ml
2,6-DA-4-NT	80 ug/ml
3,5-DNA	80 ug/ml
MNX	80 ug/ml
TNX	80 ug/ml
4,4'-TN-AZOXY	80 ug/ml

3.3.7. Special Matrix spiking solution for MNX in methanol:

MNX	80 ug/ml

4. EQUIPMENT

4.1. Magnetic stirrer with stirring bar

4.2. Standard laboratory glassware to include:

4.2.1. 1000 ml volumetric flask and various sizes of graduated cylinders

4.3. All glassware to be rinsed as follows, prior to use:

4.3.1. Technical grade acetone (if the glassware is wet).

4.3.2. Triple rinsed with methylene chloride.

4.4. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

4.5. PTFE 0.45μm syringe filter.

5. QUALITY CONTROL

QUALITY CONTROL - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction

6.1.1. Bring sample to room temperature and mix thoroughly in its original container.

6.1.2. Measure 770 mls sample into a 1000 ml volumetric flask placed on a magnetic stirrer.

6.1.3. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.4. Prepare a blank and blank spike with DIW.

6.1.5. Pipet 50 ul surrogate solution to each flask.

6.1.6. Pipet 250 ul matrix spiking solution to each of the QC flasks. (When indicated, use 250 ul special matrix spiking solution.)

6.1.7. Add 251.3 gm sodium chloride to the flask with the stirring bar at maximum speed and stir until sodium chloride is completely dissolved.

6.1.8. Add 164 mls acetonitrile to each flask and stir for 15 minutes.

6.1.9. Turn off the stirrer and let set for 10 minutes.

6.1.10. Remove acetonitrile with a Pasteur pipet and transfer to a 100 ml flask.

6.1.11. Add 10 ml acetonitrile to the sample and stir for 15 minutes.

6.1.12. Turn off stirrer and let set for 10 minutes.

6.1.13. Transfer acetonitrile to flask with extract from step 6.1.10. Discard sample in 1000 ml flask.

6.1.14. Add 84 mls salt water to extract in flask.

6.1.15. Stir for 15 minutes and let stand for 10 minutes.

6.1.16. Transfer acetonitrile to a 10 ml graduated cylinder.

6.1.17. Add 1.0 ml acetonitrile to flask and repeat steps 6.1.15 and 6.1.16.

6.1.18. Record total final volume to 0.1 ml.

6.1.19. Transfer final extract to a 4.0 ml **amber** vial. Any remaining extract may be discarded. If extract is cloudy or turbid, filter through a PTFE 0.45µm syringe filter into amber vial.

6.1.20. Label the extract and deliver to 940.

6.1.21. Complete all paperwork and bench sheet. Bench sheet to include date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be red and the blank name will be ____HOR.WL_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846,

8330A (1998).

Neymen

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 0101514 MATRIX: H_2O	8330	1-30-01	8330A	BJ	BJ 1-30-01	8330A

Sample #	wt./vol.	Final vol	To 940				SDG #	Bottle #
0101514-1	470 ml	4.0 ml	~4.0 ml				CH010	36
2		4.9						38
3		5.3						40
4		7.9						42
5		6.4						44
SMS		8.2						45
SMSD		7.6						46
S013001HORWLC		5.4						
B013001HORWLC		4.9						

1-30-01

BJ

COMMENTS:

Ext & Final Sol: Acetonitrile - Bag # BZ347

SURROGATES:

50 ul of: Ex 8.42-1A

ul of:

SPIKES:

250 ul of: Ex 8.40-10

ul of:

STANDARDS:

ul of:

ul of:

ul of:

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
8330/H_2O	Time out: 0730	Time in: 1130	Time out: 0825	Time in: —	Tray # 2-148	Time out	Time in
	Date: 1-30-01	Initials: BJ	Date: 1-31-01	Initials: AGH	F1-F9	Date	Initials
	Time out	Time in	Time out	Time in		Time out	Time in
	Date	Initials	Date	Initials		Date	Initials

Extraction Method for Ordnance Compounds in Soils (8330A)

Method # LTL-3161

May 4, 2001

Revision Number: #5

Written by: B. O Quiles Date: 5.4.01

Reviewed by: Harry Pombey Date: 9-13-01

Approved by: Gatry Grieps Date: 9-13-01

Controlled Document

No. 20 Assigned to: Tetr

1. **PURPOSE** - In this method the ordnance compounds are extracted from neutral soil with acetonitrile. The sample is air dried, sonicated, and extract salted-out and analyzed by HPLC.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2 . All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. Ottawa Sand - prepared by soxhleting for 8 hours with methylene chloride and muffling for four hours at 400°C.

3.3.2. 0.045 M Calcium Chloride - prepared by dissolving 5.0 gm in 1000 ml of DIW.

3.3.3. Surrogate solution prepared in methanol:

1,2-Dinitrobenzene	400 ug/ml

3.3.4. Matrix spiking (MS) solution prepared in methanol:

1,3-Dinitrobenzene	80 ug/ml
2,4-Dinitrotoluene	80 ug/ml
2,6-Dinitrotoluene	80 ug/ml
HMX	80 ug/ml
RDX	80 ug/ml
Nitrobenzene	80 ug/ml
2-Nitrotoluene	80 ug/ml
3-Nitrotoluene	80 ug/ml
4-Nitrotoluene	80 ug/ml
Tetryl	80 ug/ml
TNT	80 ug/ml
1,3,5-Trinitrobenzene	80 ug/ml
2-amino-4,6-dinitrotoluene	80 ug/ml
4-amino-2,6-dinitrotoluene	80 ug/ml

3.3.5. Special Matrix spiking solution for PETN/NG in methanol:

PETN	–	50 ug/ml
Nitroglycerin		100 ug/ml

3.3.6. Special Matrix spiking solution for MNX in methanol:

MNX	80 ug/ml

4. EQUIPMENT

4.1. Ultrasonic bath

4.2. #30 mesh sieve

4.3. Disposable cartridge filters - 0.45 µm Teflon filter

4.4. Standard laboratory glassware to include:

4.4.1. 20 x 150 mm culture tubes

4.5. Volumetric measurements are to be made with graduated serological pipets or a calibrated fixed volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container if there is space available (otherwise mix in solvent rinsed aluminum tray).

6.1.2. Weigh approximately 20-30 grams into a labeled, large aluminium weighing tin. Place in hood and air dry for 48 hours or until a constant weight is attained.

6.1.3. Grind and sieve.

6.1.4. Weigh 2.0 grams of air dried soil into a 20 x 150 mm culture tube.

6.1.5. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.6. Prepare a blank and blank spike with 2.0 grams Ottawa sand.

6.1.7. Pipet 100 ul surrogate solution to each culture tube.

6.1.8. Pipet 500 ul matrix spiking solution to each of the QC culture tubes.

6.1.9. Add 9.9 ml acetonitrile to each sample culture tube and blank and 9.4 mls to QC and blank spike.

6.1.10. Sonicate the samples for 18 hours in a sonic bath packed with ice in the cold room.

6.2. Salting Out

6.2.1. Remove samples from ice bath and allow to come to room temperature.

6.2.2. Transfer 5.0 mls of supernatant to a 16 x 100 mm culture tube.

6.2.3. Add 5.0 ml calcium chloride solution to each sample.

6.2.4. Shake and let stand for 15 minutes.

6.2.5. Centrifuge each sample.

6.2.6. Filter through a 0.45μm PTFE syringe filter into 4 ml **amber** vial, discarding first 3 mls.

6.2.7. Label the extract and deliver to 940.

6.3.8. Complete all paperwork and bench sheet. Bench sheet to include PTFE filter lot #, date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be red and the blank name will be ____HOR.SM_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

8330A (1998)

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 0105180	8330	5-4-01	8330A	SD	SD 5-3-01	8330A
MATRIX: Soil						

Sample #	wt./vol.	7.vap #1	Add To CaCl2	7.vap #2	Add To 940		SDG#	Bkt#
0105180-1	2.0 g	10.0 ml	5.0 ml	10.0 ml	~40 ml		T5901	28
2								29
3								30
4								31
5								32
5MS								
5MSD								
6								35
7								36
8								37
S050401HORSMG								
B050401HORSMG								

SD 5-4-01

COMMENTS:

Ext Sol: Acetonitrile - B&J # CB512
Final Sol: Acetonitrile - B&J # CB512 / 0.5% CaCl2
Samples airdried prior to extraction
Sonic Bath - On - 1545 - 5-4-01
Off - 1000 5-5-01

SURROGATES:
100 ul of: Eq 8.49-6
ul of:

SPIKES:
500 ul of: Eq 8.49-6
ul of:

STANDARDS:
ul of:
ul of:
ul of:

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
8330	Time out 1130	Time in 1305	Time out 1520	Time in —	Tray # 2-163 D1-D12	Time out	Time in
	Date 5-3-01	Initials SD	Date 5-6-01	Initials SD		Date	Initials
	Time out	Time in	Time out —	Time in 1531		Time out	Time in
	Date	Initials	Date 5-6-01	Initials SD		Date	Initials

Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081A/8082 by 3510C)

Method # LTL-3202

May 7, 2001

Revision Number: #3

Written by: _____ Date: 5-7-01

Reviewed by: Harry Lombey Date: 5-11-01

Approved by: Kathy Kreps Date: 5-11-01

Controlled Document

No. 20 Assigned to: TETR

1. **PURPOSE** - In this method, organochlorine pesticides and polychlorinated biphenyls are extracted from neutral water with methylene chloride. The extracts are dehydrated, concentrated in a Kuderna-Danish apparatus and exchanged into hexane. If necessary, the pesticide/PCB extracts may be SPE Florisil cleaned prior to GC analysis. PCB only extracts may be acid cleaned prior to GC analysis.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2. All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. 10 N Sodium Hydroxide - weigh 400 grams sodium hydroxide, dissolve and dilute to one liter with DIW.

3.3.2. 1:1 Sulfuric Acid - measure equal volumes of concentrated sulfuric acid and DIW. Note: Observe proper acid preparation techniques.

3.3.3. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.4. Surrogate solution prepared in acetone:

2, 4, 5, 6-Tetrachloro-m-xylene	1.0 ug/ml
Decachlorobiphenyl	1.0 ug/ml

3.3.5. Pesticide and/or PCB matrix spiking solution prepared in acetone:

Gamma-BHC	2.5 ug/ml
Heptachlor	2.5 ug/ml
Aldrin	2.5 ug/ml
Arochlor 1260	25.0 ug/ml

4. **EQUIPMENT**

4.1. Glas-Col mechanical separatory funnel shaker

4.2. Organomations Assoc., Inc. - N-EVAP, (Nitrogen Evaporator), Model 112

4.3. Vortex mixer

4.4. Standard laboratory glassware to include:

4.4.1. 2000 ml Teflon separatory funnels

4.4.2. 300 ml Fleakers

4.4.3. K-D apparatus: 250 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.5. All glassware to be rinsed as follows, prior to use:

4.5.1. Technical grade acetone (if the glassware is wet).

4.5.2. Triple rinsed with methylene chloride.

4.6. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blank, blank spikes matrix spike and matrix spike duplicate (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container and place labeling tape at sample level. Mark meniscus of sample on the tape.

6.1.2. Transfer entire contents of container into a 2000 ml separatory funnel. Rinse container with methylene chloride and add to funnel. Fill sample container to the mark on tape with water. Measure volume of sample used in a calibrated 1000 ml cylinder.

6.1.3. Prepare a blank and a blank spike with 1000 ml DIW.

6.1.4. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.5. Pipet 200 ul surrogate solution into each funnel.

6.1.6. Pipet 200 ul matrix spiking solution into each QC funnel.

6.1.7. Adjust pH to 7 ± 2.

6.1.8. Add 60 ml methylene chloride to the separatory funnel.

6.1.9. Shake the samples hard for three minutes on the mechanical shaker, with the stopcocks closed.

6.1.10. Replace the funnels in rack and allow to stand for 10 minutes.

6.1.11. Drain and collect the lower methylene chloride layer in a fleaker.

6.1.12. Repeat from 6.1.8. until a total of three extractions have been performed.

6.2. Solvent Dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from Step 6.1.12. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent Evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 8 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml, and exchange into hexane as follows:

6.3.3.1. Add 2-3 ml hexane through the top of the snyder column while the ampule is still immersed in the water bath. Reduce to 4-5 ml.

6.3.3.2. Repeat 6.3.3.1. two additional times.

6.3.4. Remove the apparatus from the water bath and cool to room temperature.

6.3.5. Rinse joint and remove the snyder column. Allow rinse solvent to drain into ampule.

6.3.6. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of hexane.

6.3.7. Reduce the extract volume to less than 2 ml on a nitrogen blowdown. Transfer extract to a 16 x 100 mm culture tube, and adjust to 10.0 ml final volume (as compared to a measured volume) with hexane.

6.3.8. Label the extract and deliver to 940.

6.3.9. Complete all necessary paperwork and bench sheet. Bench sheet to include cleanup method #, if applicable, extract location, time and date of transfer to 940. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be purple and the blank name will be ____GPXWL_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846, (1996).

8081A, 8082, 3510C

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 0101862	Pest / PCB	1-30-01	3510C	LS	BD 1-31-01	8081A / 8082
MATRIX: H2O						

Sample #	wt./vol.	F. vol					SDG #	Std #
0101862-1	1020 mL	10.0 mL					LA 492	1
1 MS	1000							2
1 MSD	1060							3
2	980							5
3	1050							7
4	1030							9
5	1040							11
B013001GPX WLG	1000							
B013001GPX WLG	1	1						

1-30-01 BD/LS

COMMENTS:

Ext Solv: Methylene - BEJ # BZ743
Final Solv: Hexane - EM # 40163

SURROGATES:

200 ul of: Ex 8-45-9A
_____ ul of: _____

SPIKES:

200 ul of: Ex 8-39-4
_____ ul of: _____

STANDARDS:

_____ ul of: _____
_____ ul of: _____
_____ ul of: _____

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
Pest / PCB	Time out: 0910	Time in: Empty	Time out: 1550	Time in: —	Tray # 2-15-01 B1-B9	Time out:	Time in:
	Date: 1-30-01	Initials: LS	Date: 2-1-01	Initials: AGH		Date:	Initials:
	Time out:	Time in:	Time out: —	Time in: 1555		Time out:	Time in:
	Date:	Initials:	Date: 2-1-01	Initials: SD		Date:	Initials:

Extraction Method for Chlorinated Herbicides in Water
(8151A by Modified 3510C)

Method # LTL-3510

May 3, 2001

Revision Number: #8

Written by: B. O Gill Date: 5-3-01

Reviewed by: Harry Rumbley Date: 5-3-01

Approved by: Kathy Gregg Date: 05-03-01

Controlled Document

No. 20 Assigned to: TETR

1. **PURPOSE** - In this method, samples are first subjected to an alkaline hydrolysis. The sample is extracted while alkaline to remove extraneous materials. It is then acidified, forming the less polar species, which are extracted into ethyl ether. The extracts are derivatized with diazomethane and the methyl esters are isolated for analysis by GC/ECD.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples, by their very nature, may contain significant levels of hazardous materials. It is incumbent on each analyst to exercise due care and caution executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **MATERIALS**

3.1. All reagents are to be AR grade or better.

3.2. All solvents are to be distilled in glass unless otherwise noted.

3.3 The following special reagents should be prepared:

3.3.1. 37% Potassium Hydroxide - prepared by dissolving 370 grams potassium hydoxide pellets and diluting to one liter with DIW.

3.3.2. Concentrated Sulfuric Acid

3.3.3. 10% Hydrochloric Acid - measure 100 ml concentrated hydrochloric acid and dilute to 1000 ml with DIW. Note: Observe proper acid preparation techniques.

3.3.4. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.5. Acidified Sodium Sulfate - Weigh the muffled anhydrous sodium sulfate in 100 gram increments into a suction flask. Cover the sodium sulfate with ethyl ether containing 0.1 ml concentrated sulfuric acid for each 100 g increment. Swirl the slurry for about two minutes then stopper the flask and apply an aspirator vacuum until the ether is completely removed . Place on a steam bath in a hood to assure that all ether is evaporated. Store in a closed bottle.

3.3.6. 10 M Potassium Hydroxide - prepared by dissolving 56.1 grams potassium hydoxide pellets and diluting to 100 ml with DIW.

3.3.7. Diethylene Glycol Monoethyl Ether (Carbitol) – (Sigma – catalog # D-1265)

3.3.8. Diazald® (Aldrich - catalog # D2,800-0).

3.3.9. Surrogate solution prepared in methanol:

2,4 - Dichlorophenyl Acetic Acid	5.0 ug/ml

3.3.10. Matrix Spiking solution prepared in methanol:

2,4,5-TP	0.8 ug/ml
2,4,5-T	0.8 ug/ml
Dicamba	0.8 ug/ml
Pentachlorophenol	4.0 ug/ml
Dinoseb	4.0 ug/ml
Picloram	8.0 ug/ml
2,4-DB	8.0 ug/ml
2,4-D	8.0 ug/ml
Dichloroprop	8.0 ug/ml
Dalapon	20.0 ug/ml
MCPP	800.0 ug/ml
MCPA	800.0 ug/ml

4. EQUIPMENT

4.1. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.2. Standard laboratory glassware to include:

4.2.1. 25 x 200 mm Pyrex culture tubes

4.2.2. 16 x 100 mm culture tubes

4.3. All glassware to be used shall be rinsed in the following manner:

4.3.1. 10% hydrochloric acid

4.3.1. Reagent grade acetone

4.4. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blank, blank spike, matrix spike and matrix spike duplicate (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. METHODOLOGY

6.1. Sample Extraction:

6.1.1. Mix sample thoroughly in its original container.

6.1.2. Measure 50 ml sample into 25 x 200 culture tube.

6.1.3. Prepare a blank and a blank spike with 50 ml DIW.

6.1.4. Prepare two additional aliquots of a sample if Quality Control is required.

6.1.5. Pipet 10 ul surrogate solution into each tube.

6.1.6. Pipet 10 ul matrix spiking solution into each QC tube.

6.1.7. Pipet 1.0 ml 37% potassium hydoxide into each culture tube, mix and loosely cap with teflon screw cap.

6.1.8. Immerse tubes in a 60-65°C water bath for a minimum of 90 minutes.

6.1.9. Remove from bath and cool to room temperature.

6.1.10. Add 6-7 mls ethyl ether to each culture tube, cap tightly and shake for two minutes. If emulsions occur, centrifuge tubes.

6.1.11. Draw off and discard ether layer.

6.1.12. Repeat from step 6.1.10. two additional times using 3 ml ethyl ether.

6.1.13. Adjust pH to <2 by slowly adding 500µl concentrated sulfuric acid to each tube. Mix and check pH.

6.1.14. Repeat step 6.1.10 three times using 3 ml ethyl ether and save ether into a 16 x 100 culture tube.

6.2. Dehydration of the extract:

6.2.1. Adjust volume to 5-6 mls in the nitrogen blowdown.

6.2.2. Add 3 gm of acidified sodium sulfate to each tube, cap and shake.

6.2.3. Tap the tubes to settle the sodium sulfate and let stand at least 60 minutes and may stand overnight.

6.2.4. Transfer ether extract to 16 x 100 culture tube.

6.2.5. Add 1-2 mls ether to acidified sodium sulfate, cap and shake to break up sodium sulfate.

6.2.6. Transfer rinse to tube containing the ether extract.

6.2.7. Repeat from step 6.2.5. two additional times.

6.2.8. Adjust volume to 5-6 mls in the nitrogen blowdown.

6.3. Derivatization:

6.3.1. To tube A (the first tube in the train), add 1.0 ml methanol, and bring to 8.0 ml with ethyl ether.

6.3.2. To tube B add:

6.3.2.1. 2.0 ml 10 M potassium hydoxide (to the first marked line)

6.3.2.2. 1.4 ml Carbitol (diethylene glycol monoethyl ether) to the second marked line.

6.3.3. Place 10 ul micro-pipet in each sample tube from step 6.2.8.

6.3.4. Connect nitrogen line to the apparatus and regulate so that tube A bubbles actively but not violently.

6.3.5. Add 2 scoops (approximately 0.1-0.2 grams) of Diazald and 1.4 ml ethyl ether to tube B. Stopper, connecting tube A to tube B.

6.3.6. Bubble the generated diazomethane into the tube for 90 seconds. (Extract will turn bright yellow.)

6.3.7. Let extract stand for 15 minutes.

6.3.8. Reduce extract to near dryness on nitrogen blowdown. Add 5 ml hexane.

6.3.9. Reduce the extract volume to less than 0.5 ml in a nitrogen blowdown.

6.3.10. Adjust the final volume to 0.5 ml (as compared to a measured volume) with hexane.

6.3.11. Label the extract and deliver to 940.

6.3.12. Complete all the necessary paperwork and bench sheet. Bench sheet to include date derivatized, date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in the GC room extraction folder box. The file folder color will be yellow, and the blank name will be ____GHB.WL_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

8151A (1996), 3510C (1996)

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 0104123	Vesb	4-10-01	mod 3510C	YL	4-11-01	8151A
MATRIX: H_2O						

Sample #	wt./vol.	% vol					SDG#	Bottle #
0104132-1	50 ml	0.5ml					LI143	13
2								14
3								15
3MS								16
3MSD								17
4								18
5								20
6								22
S010401GHBWLG								
B010401GHBWLG								

4-10-01 YL

COMMENTS:

Ext Sol: Ethyl Ether - BGJ # CB192
Final Sol: Hexane - EM # 40163
Derivatized: 4-11-01

SURROGATES:

10 ul of: Ex 8.44-5
ul of:

SPIKES:

10 ul of: Ex 8-44-8
ul of:

STANDARDS:

ul of:
ul of:
ul of:

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
Vesb	Time out: 0900	Time in: 1000	Time out: 1350	Time in: —	Tray # 1564 A1 – A10	Time out:	Time in:
	Date: 4-10-01	Initials: YL	Date: 4-12-01	Initials: [illegible]		Date:	Initials:
	Time out:	Time in:	Time out: —	Time in: 1400		Time out:	Time in:
	Date:	Initials:	Date: 4-12-01	Initials: SO		Date:	Initials:

Extraction Method for Picric and Picramic Acids in Water
(3510C)

Method # LTL-3550

April 12, 2001

Revision Number: #5

Written by: B. O'Neill Date: 4-12-01

Reviewed by: Harry Romley Date: 4-26-01

Approved by: Kathy Krepp Date: 04-30-01

Controlled Document

No. 20 Assigned to: TETR

1. **PURPOSE:** In this method, the ion pairing reagent, tetrabutylammonium dihydrogen phosphate (TBAP), is added to a water sample, pH adjusted to slightly basic and extracted with methylene chloride. Extracts are dehydrated and concentrated in a Kuderna-Danish (K-D) apparatus and exchanged to methanol prior to analysis by HPLC.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2 . All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. 10 N Sodium Hydroxide - prepared by weighing 400 grams sodium hydroxide, dissolve and dilute to one liter with DIW.

3.3.2. 1:1 Sulfuric Acid - measure equal volumes of concentrated sulfuric acid and DIW. Note: Observe proper acid preparation techniques.

3.3.3. Tetrabutylammonium Dihydrogen Phosphate (TBAP) - prepared by weighing 1.0 gm TBAP, dissolve and dilute to one liter with DIW.

3.3.4. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate four hours at 400°C.

3.3.5. Surrogate Solution prepared in methanol:

4,6-Dinitro-*o*-cresol	80.0 ug/ml

3.3.6. Matrix Spiking Solution prepared in methanol:

Picric Acid	40.0 ug/ml
Picramic Acid	40.0 ug/ml

4. **EQUIPMENT**

4.1. Glas-Col mechanical separatory funnel shaker

4.2. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.3. Standard laboratory glassware to include:

4.3.1. 2000 ml Teflon separatory funnels

4.3.2. 300 ml Fleakers

4.3.3. K-D apparatus: 250 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.4. All glassware to be rinsed as follows, prior to use:

4.4.1. Technical grade acetone (if the glassware is wet).

4.4.2. Triple rinsed with methylene chloride.

4.5. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blank, blank spike, matrix spike and matrix spike duplicate (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container and place labeling tape at sample level. Mark meniscus of sample on the tape.

6.1.2. Transfer entire contents of container into a 2000 ml separatory funnel. Rinse container with methylene chloride and add to funnel. Fill sample container to the mark on tape with water. Measure volume of sample used in a calibrated 1000 ml cylinder.

6.1.3. Prepare a blank and a blank spike with 1000 ml DIW.

6.1.4. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.5. Add 100 ml TBAP solution to each funnel.

6.1.6. Pipet 100 ul surrogate solution into each funnel.

6.1.7. Pipet 100 ul matrix spiking solution into each QC funnel.

6.1.8. Adjust pH to 8.5 - 9.

6.1.9. Add 60 ml methylene chloride to each separatory funnel.

6.1.10. Shake the samples hard for three minutes on the mechanical shaker, with the stopcocks closed.

6.1.11. Replace the funnels in rack and allow to stand for 10 minutes.

6.1.12. Drain and collect the lower methylene chloride layer in a Fleaker.

6.1.13. Repeat from 6.1.9. until a total of three extractions have been performed.

6.2. Solvent dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with muffled sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from 6.1.13. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 8 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml, and exchange into methanol as follows:

6.3.3.1. Add 2-3 ml methanol through the top of the snyder column while the ampule is still immersed in the water bath. Reduce to 4-5 ml.

6.3.3.2. Repeat 6.3.3.1. two additional times.

6.3.4. Remove the apparatus from the water bath and cool to room temperature.

6.3.5. Rinse joint with methanol and remove the snyder column. Allow rinse solvent to drain into ampule.

6.3.6. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of methanol.

6.3.7. Reduce the extract volume to below 0.5 ml in a warm water bath with nitrogen to insure that there has been a complete exchange to methanol. Rinse internal walls of ampule several times during blowdown.

6.3.8. Transfer extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methanol.

6.3.9. Label the extract and deliver to 940.

6.3.10. Complete all paperwork and bench sheet. Bench sheet to include date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be neon green and the blank name will be ____HSV.WL_.

7. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

3510C (1996)

LAUCKS TESTING LABORATORIES INC.	EXTRACTION	DATE	METHOD	ANALYST	CHECKER	ANALYSIS
JOB NUMBER: 0104217	Pic/Pic	4/12/01	3510C	LS.	LS 4/12/01	Picric/ Picramic
MATRIX: H_2O						

SDG LE001

Sample #	wt./vol.	Final Vol.						Btl #
0104217-1	1000ml	1.0ml						1
-2								3
-2ms								4
-2msD								5
-3								9
-4								11
-5								13
-6								15
S041201HSVWLS								
B041201HSVWLS								

4/12/01 LS.

COMMENTS:

- Ext. Solvent: MeCl2 B+J CA561
- Final Solvent: MeOH Em # 39022
- pH 8.5-9

SURROGATES:

100 ul of: EXB-39-5

ul of:

SPIKES:

100 ul of: EXB-43-4

ul of:

STANDARDS:

ul of:

ul of:

ul of:

Fraction and Method	Extraction		Transferred from 921 to 940		Extract Location	Analysis	
Pic/Pic-H_2O	Time out: 0900	Time in: Empty	Time out: 1600	Time in: —	Tray # 1565 AA0-AA9	Time out:	Time in:
	Date: 4/12/01	Initials: LS.	Date: 4/12/01	Initials: AGH		Date:	Initials:
	Time out:	Time in:	Time out: —	Time in: 1610		Time out:	Time in:
	Date:	Initials:	Date: 4/12/01	Initials: SO		Date:	Initials:

Extraction Method for Picric and Picramic Acids in Soil (3550B)

Method # LTL-3555

November 29, 1999

Revision Number: #4

Written by: B. O'Neill Date: 11-29-99

Reviewed by: Harry Rombery Date: 12-13-99

Approved by: Kathy Grieps Date: 12-13-99

Controlled Document

No. 20 Assigned to: Tetra

1. **PURPOSE:** In this method, picric/picramic acids are extracted from soil with 80:20 DIW/methanol. Ion pairing reagent, tetrabutylammonium dihydrogen phosphate (TBAP), is added to the water sample, pH adjusted to slightly basic and extracted with methylene chloride. Extracts are dehydrated, concentrated in a Kuderna-Danish (K-D) apparatus and exchanged to methanol prior to analysis by HPLC.

2. **SAFETY** - During the conduct of this method, the extractionist will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **REAGENTS**

3.1. All reagents shall be of AR grade or better.

3.2. All solvents shall be distilled in glass unless otherwise indicated.

3.3. The following special reagents shall be prepared:

3.3.1. 10 N Sodium Hydroxide - prepared by weighing 400 grams sodium hydroxide, dissolve and dilute to one liter with DIW.

3.3.2. 1:1 Sulfuric Acid - measure equal volumes of concentrated sulfuric acid and DIW. Note: Observe proper acid preparation techniques.

3.3.3. Tetrabutylammonium Dihydrogen Phosphate (TBAP) - prepared by weighing 1.0 gm TBAP, dissolve and dilute to one liter with DIW.

3.3.4. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate four hours at 400°C.

3.3.5. Surrogate solution prepared in methanol:

4,6-Dinitro-o-cresol	80.0 ug/ml

3.3.6. Matrix Spiking solution prepared in methanol:

Picric Acid	40.0 ug/ml
Picramic Acid	40.0 ug/ml

4. **EQUIPMENT**

4.1. Heat Systems Ultrasonic Processor - Model XL2020, 550 watts - Maintain per manufacturer's instructions.

4.1.1. 3/4 inch titanium horn (#208).

4.2. Organomations Assoc., Inc. - N-EVAP, (nitrogen evaporator), Model 112

4.3. Standard laboratory glassware to include:

4.3.1. 8 ounce extraction bottles

4.3.2. 300 ml Fleakers

4.3.3. 1000 ml Teflon separatory funnels

4.3.3. K-D apparatus: 250 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.4. All glassware to be rinsed as follows, prior to use:

4.4.1. Technical grade acetone (if the glassware is wet).

4.4.2. Triple rinsed with methylene chloride.

4.5. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container.

6.1.2. Weigh 30.0 grams of soil into an extraction bottle.

6.1.3. Prepare two additional aliquots of one sample if Quality Control is required.

6.1.4. Prepare a blank and blank spike with 60 grams sodium sulfate.

6.1.5. Pipet 100 ul surrogate solution to each bottle.

6.1.6. Pipet 100 ul matrix spiking solution to each of the QC bottles.

6.1.7. Add 80 ml DIW to each extraction bottle.

6.1.8. Adjust pH to 8.5 - 9.0.

6.1.9. Add 20 ml methanol to each bottle.

6.1.10. Sonicate the bottles for 3 minutes using the sonic horn, set at 50% duty cycle and full output (10).

6.1.11. Centrifuge the samples for 20 minutes at 2000 rpm.

6.1.12. Decant off and collect the supernatant in a 1000 separatory funnel.

6.1.13. Adjust pH to 8.0 - 8.5.

6.1.14. Add 100 ml TBAP solution.

6.1.15. Add 60 ml methylene chloride to each separatory funnel.

6.1.16. Shake the samples hard for three minutes on the mechanical shaker, with the stopcocks closed.

6.1.17. Replace the funnels in rack and allow to stand for 10 minutes.

6.1.18. Drain and collect the lower methylene chloride layer in a Fleaker.

6.1.19. Repeat from step 6.1.15. until a total of three extractions have been performed.

6.2. Solvent dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with muffled sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from step 6.1.19. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 8 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml, and exchange into methanol as follows:

6.3.3.1. Add 2-3 ml methanol through the top of the snyder column while the ampule is still immersed in the water bath. Reduce to 4-5 ml.

6.3.3.2. Repeat 6.3.3.1. two additional times.

6.3.4. Remove the apparatus from the water bath and cool to room temperature.

6.3.5. Rinse joint with methanol and remove the snyder column. Allow rinse solvent to drain into ampule.

6.3.6. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of methanol.

6.3.7. Reduce the extract volume to below 0.5 ml in a warm water bath with nitrogen to insure that there has been a complete exchange to methanol. Rinse internal walls of ampule several times during blowdown.

6.3.8. Transfer extract to a 1.8 ml vial. The final volume is then adjusted to 1.0 ml (as compared to a measured volume) in methanol.

6.3.8. Label the extract and deliver to 940.

6.3.9. Complete all paperwork and bench sheet. Bench sheet to include date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be neon green and the blank name will be ____HSV.SL_.

7. **ANALYSIS TIME** - Based on experience in the laboratory, it is anticipated that a single sample may be completed in about six hours. If it is possible to batch similar samples, it is expected that about ten samples could be completed in approximately 18 hours. About one hour of actual hands on time will be expended by the extractionist. These approximate times are based on the assumption that the samples are "average", and will not require additional time beyond normal operations. Additional time must be allocated for samples which are very dirty or are extraordinary.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

3550B (1996), 3510C (1996)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-6006

Title: Cation-Exchange Capacity of Soils (Sodium Acetate) by SW 846 Method 9081

Revision history:

Number	Date
0	1/25/99

UNCONTROLLED

Written by: Harry Romberg Date: 1-25-99
Harry Romberg, Quality Assurance Officer

Reviewed by: Raul N Date: 1/25/99
Raul Nino, Analyst

Approved by: Kathy Kreps Date: 1/25/99
Kathy Kreps, Laboratory Director

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QC SUMMARY TABLE 8

APPENDIX II **10**

FLOW CHART 10

1. Introduction and Scope

1.1 Scope

1.1.1 LTL-6006 (SW 846 Method 9081) is applicable to most soils, including calcareous and noncalcareous soils. This method only applies to the Cation Exchange Capacity extraction. The actual analysis method should be referred to for quantitation of sodium in the extract.

1.2 Method Description

1.2.1 The soil sample is mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. Subsequently, the sample is washed with isopropyl alcohol. An ammonium acetate solution is then added, which replaces the adsorbed sodium with ammonium. The concentration of displaced sodium is then determined by flame atomic absorption spectroscopy or ICP.

1.2.2 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 No holding times or preservation are specified for this method.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.4.2 Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. This is followed by the date, analysis, matrix and sequence number (i.e. BmmddyyCECS01).

1.4.3 CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.4.4 DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.4.5 RPD - Relative percent difference, in this case between duplicate analyses, calculated as the difference between replicate sample determinations divided by the average calculated as a percent.

2. Equipment List and Standards

2.1 Equipment

- 50 mL centrifuge tubes and stoppers
- mechanical shaker
- 100 mL class A volumetric flasks

2.2 Reagents

2.2.1 Sodium acetate (NaOAc), 1.0 N: Dissolve 136 g of $NaC_2H_2O_2 \cdot 3H_2O$ (or 82 g anhydrous $NaC_2H_2O_2$) in water and dilute it to 1,000 mL. The pH of this solution should be 8.2. If needed, add a few drops of acetic acid or NaOH solution to bring the reaction of the solution to pH 8.2.

2.2.2 Ammonium acetate (NH_4OAc), 1 N: Dilute 114 mL of glacial acetic acid (99.5%) with water to a volume of approximately 1 liter. Then add 138 mL of concentrated ammonium hydroxide (NH_4OH) and add water to obtain a volume of about 1,980 mL. Check the pH of the resulting solution, add more NH_4OH, as needed, to obtain a pH of 7, and dilute the solution to a volume of 2 liters with water.

2.2.3 Isopropyl alcohol: 99%.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.2 Waste Disposal

3.2.1 Isopropyl alcohol solutions should be disposed of in the solvent waste container.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 Method Blanks

4.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the reporting limit is reported. Method blank control limits are that contamination should not exceed the reporting limit.

4.1.2 Corrective action

4.1.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 20 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.2 Sample Duplicate

4.2.1 Criteria

4.2.1.1 At least one duplicate sample per 10 samples is required. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.2.1.2 The RPD control limits are detailed in the Quality Control Database (QC_DB) and will change from time to time.

4.2.2 Corrective action

4.2.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Extraction Procedure

5.1.1 Weigh 4 g of medium or fine-textured soil or 6 g of coarse-textured soil and transfer the sample to a 50-mL, round-bottom, narrow-neck centrifuge tube. (A fine soil has >50% of the particles <0.074 mm, medium soil has >50% >0.425 mm, while a coarse soil has more than 50% of its particles >2 mm.

5.1.2 Add 33 mL of 1.0 N NaOAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

5.1.3 Decant the liquid, and repeat Paragraph 5.1.2 three more times.

5.1.4 Add 33 mL of 99% isopropyl alcohol, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear.

5.1.5 Repeat the procedure described in Paragraph 5.1.4 two more times.

5.1.6 Add 33 mL of NH_4OAc solution, stopper the tube, shake it in a mechanical shaker for 5 min, and centrifuge it until the supernatant liquid is clear. Decant the washing into a 100-mL volumetric flask.

5.1.7 Repeat the procedure described in Paragraph 5.1.6 two more times.

5.1.8 Dilute the combined washing to the 100-mL mark with ammonium acetate solution and determine the sodium concentration by atomic absorption, emission spectroscopy, or an equivalent method.

5.2 Instrumental Analysis

5.2.1 Specifics of the instrumental analysis are contained in the appropriate SOP.

6. Reports

6.1 Data Packet Organization

6.1.1 Sample reports vary depending upon the needs of the client. They may be as simple as a standard laboratory report with only a QC summary (generated from QC_DB) or they may be more thorough and include instrument calibration information and raw data if a full data package is required.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered must be included in the data packet. The routine minimum for this analysis is a method blank report and a duplicate report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

Appendix I

QC Summary Table

Laucks Testing Laboratories

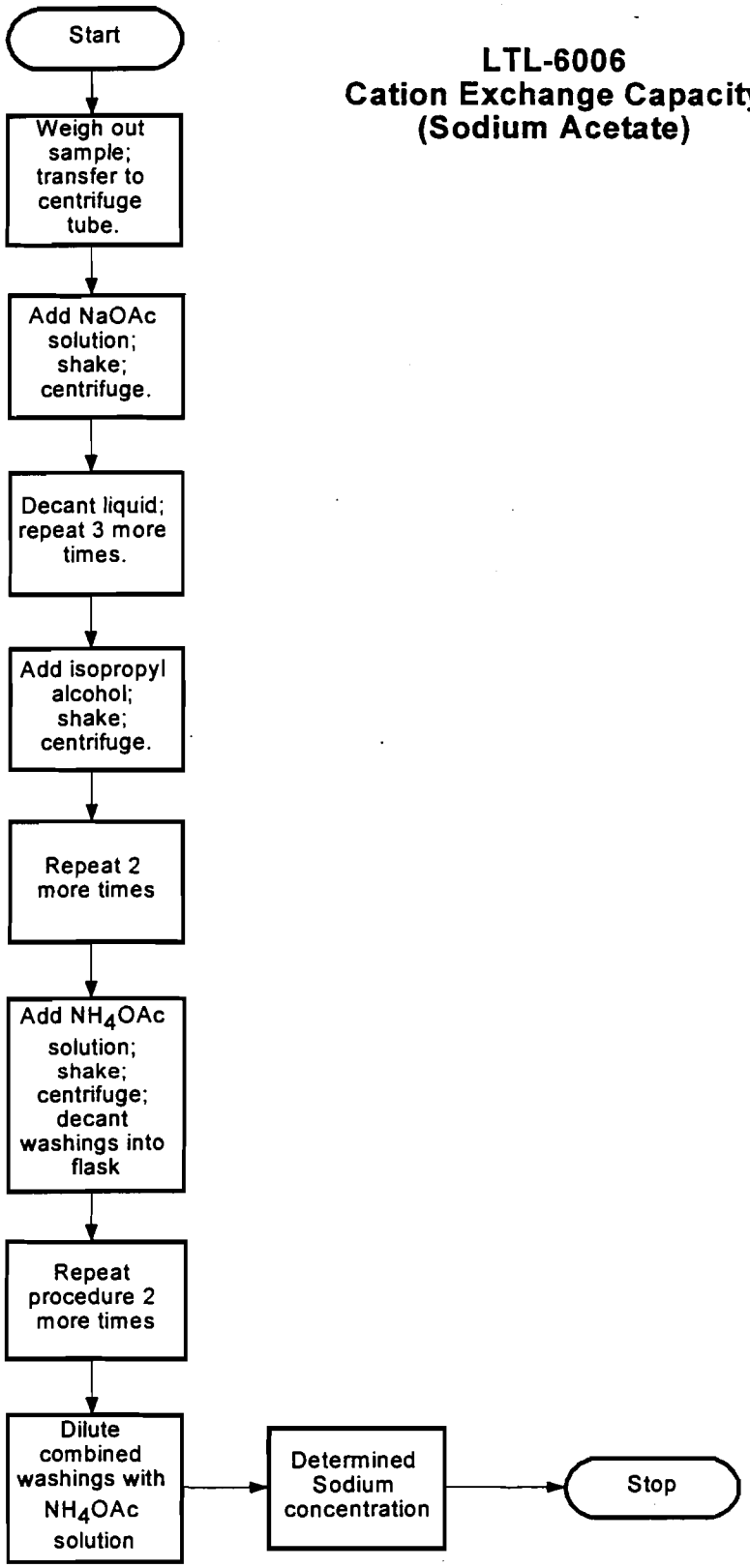
Method LTL-6006 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	None	No blank above reporting limit	1 per 20 samples or one per batch	reanalyze unless samples >20X blank	QC_DB and sample report
Duplicate % Difference or RPD	None	See QC_DB database	1 per 10 samples or one per batch	Discuss with QC Officer	QC_DB and sample report

Appendix II

Flow Chart

LTL-6006
Cation Exchange Capacity
(Sodium Acetate)



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-6008

Title: **Determination of Acid Volatile Sulfides (AVS) and Simultaneously Extractable Metals (SEM) in Sediment**

Revision history:

Number	Date
0	10/03/01

Written by: Raul J. Nino, Chemist Date: 10/10/01

Approved by: Harry Romberg, QA Officer Date: 10-8-01

Approved by: Kathy Kreps, Laboratory Director Date: 10-8-01

Controlled Document

No. 20 **Assigned to:** Tetr

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1. Scope, Summary of Method, Interferences, and Definitions

1.1 Scope and Application

1.1.1 This method describes procedures for the determination of acid volatile sulfides (AVS) and for metals that are solubilized during the acidification step (simultaneously extracted metal, SEM). The conditions used have been reported to measure amorphous or moderately crystalline monosulfides. As a precipitant of toxic heavy metals, sulfide is important in controlling the bioavailability of metals in anoxic sediments. If the molar ratio of toxic metals measured by SEM to AVS exceeds one, the metals are potentially bioavailable. Because the relative amounts of AVS and SEM are important in the prediction of potential metal bioavailability, it is important to use the SEM procedure for sample preparation for metal analysis. This uses the same conditions for release of both sulfide and metal from the sediment and thus provides the most predictive means of assessing the amount of metal associated with sulfide.

1.2 Summary of Method

1.2.1 The AVS in the sample is first converted to hydrogen sulfide (H_2S) gas by acidification with hydrochloric acid at room temperature. The H_2S is then purged from a sample and trapped as silver sulfide (Ag_2S). The amount of Ag_2S formed is determined gravimetrically, and calculated as µmoles/g DB of AVS.

1.2.2 The SEM part of the analysis is determined after the H_2S gas has been released from the sample in the reaction vessel. The acidified solution is then filtered and analyzed by inductive coupled plasma (ICP) for metals, and reported as µmoles/g DB for each metal.

1.2.3 The sum total of all the metals found in the SEM analysis divided by the sulfide found in the AVS analysis is then used to determine the bioavailablity of the sample, reported as µmoles/g of SEM/AVS .

1.3 Interferences

1.3.1 Oxygen in the reagents and apparatus is the primary interference reported. Special precautions must be taken to insure that the analytical system is adequately purged with oxygen-free nitrogen.

1.3.2 The pH of the sample after the addition of the acid and during the purge process must be below 3. Typically, the pH is below 2.

1.4 Sample Collection, Preservation and Storage

1.4.1 The samples should be collected in a wide mouth jar with minimum air space above the sediment and sealed with a Teflon lined lid or a polyethylene lined lid.

1.4.2 If possible, the headspace should be purged with oxygen free nitrogen, otherwise there are no other sample preservation required.

1.4.3 Samples should be cooled to 4°C as soon as possible after collection and be kept at 4°C until sample analysis. Holding time for the samples should not exceed 14 days from the date of collection.

1.5 Definition of Terms

1.5.1 This section defines terms and acronyms as they are used in this SOP.

Acid Volatile Sulfides (AVS)	Amorphous, moderately crystalline monosulfide, and other sulfides that form hydrogen sulfide under the conditions of this test.
Simultaneously Extracted Metals (SEM)	Metals, commonly cadmium, copper, lead, mercury, nickel and zinc, which form less soluble sulfides than do iron or manganese, and which are at least partially soluble under the conditions of this test.
DIW	Deionized water. Lab reagent water.

2. Safety precautions and Waste Disposal

2.1 Routine Safety Precautions

2.1.1 All samples and solutions should be handled as if they contain hazardous substances.

2.1.2 Some of the reagents used may causes irritation and/or be harmful if swallowed or inhaled. Read the MSDS for proper handling and storage of the reagents used.

2.1.3 Hydrogen sulfide is a highly poisonous, gaseous compound having a characteristic odor of rotten eggs. It is detectable in air by humans at a concentration of 0.002 ppm. However, the human nose quickly adjusts to the presence of sulfide up to the point that it can actually become hazardous. Thus, ventilation is necessary and caution must be exercised.

2.1.4 Refer to the instrument manufacturer's manual for routine instrument precautions.

2.1.5 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component and have the potential to do harm if not used properly.

2.1.6 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully

2.1.7 Grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

2.2 Waste disposal

2.2.1 Any unused or used acidic solutions must be neutralized with sodium bicarbonate prior to disposal down the sink and flushed with copious amounts of water.

2.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

3. Equipment List and Solutions

3.1 Equipment List

3.1.1 Assorted Calibrated Pipettes and Volumetric Flasks.

3.1.2 Nitrogen Gas – 99.999% pure.

3.1.3 Flow Controller.

3.1.4 250 mL Erlenmeyer Flasks.

3.1.5 Rubber Stoppers – Two and three holed.

3.1.6 125 ml Separatory Funnel.

3.1.7 Filtering Flask with 47 mm Filter Holder.

3.1.8 Pre-Weighted 47 mm Glass Fiber Filter.

3.1.9 Drying Oven – Capable of maintaining a constant temperature of 100°C ± 5°C.

3.1.10 Course (2.7 micron) Glass fiber filters (Whatman GF/D)

3.1.11 Analytical Balance – A balance sensitive to 0.0001 grams.

3.1.12 Magnetic Stirrer (thermally insulated) and Teflon-Coated Stirring Bars.

3.2 Solutions

3.2.1 Hydrochloric Acid, 6M – Dilute 500 mL of concentrated hydrochloric acid to 1 L with DIW. Purge with oxygen free nitrogen gas for 30 minutes before use.

3.2.2 Potassium Acid Phthalate, 0.05M – Dissolve 10.2 gm of potassium acid phthalate in DIW and dilute to 1 L.

3.2.3 Silver Nitrate, 1M – Dissolve 17 gm of silver nitrate in DIW and dilute to 1 L. Store in a dark bottle.

3.2.4 Starch Indicator – Dissolve 20 gm soluble starch in 1 L of boiling DIW, after the solution has cooled add 2 gm of buffered aspirin to preserve the solution.

3.2.5 Potassium Bi-iodate, 0.025N – Potassium bi-iodate solution is purchased as a pre-made certified solution. If necessary, it can be made by dissolving 0.8124 gm of potassium bi-iodate in DIW and diluting it to 1000 mL.

3.2.6 Sodium Thiosulfate, 0.025N – Dissolve 12.41 gm of sodium thiosulfate 5-hydrate into about 900 mL of DIW in a 1000 mL volumetric flask, add 1.5 mL or 0.4 gm of sodium hydroxide, and fill the volumetric flask to 1000 mL with DIW. Standardize with the potassium bi-iodate solution.

3.2.6.1 Standardization – Dissolve approximately 2 gm of potassium iodide (free from iodate) in an erlenmeyer flask with 100 to 150 mL of DIW. Add 1 mL of 6N hydrochloric acid and 20 mL of 0.025N potassium bi-iodate. Dilute to 200 mL and titrate liberated iodine with the sodium thiosulfate solution. Add starch toward the end of the titration, when a pale yellow is reached. Adding starch will make the solution dark blue to light blue, depending on how close to the end of the titration. Continue titrating until the solution is colorless. 20 mL of the sodium thoisulfate would be necessary to neutralize 20 mL of 0.025N potassium bi-iodate. If not then adjust the sodium thiosulfate solution, until they match.

3.2.7 Iodine, 0.025N – Dissolve 22.5 gm of potassium iodide, in a small volume of DIW (<100 mL). Add 3.2 gm iodine. Wait for the iodine to dissolve then dilute to 1000 mL with DIW. Standardize against the standardized 0.025N sodium thiosulfate solution before each use.

3.3 Standards

3.3.1 Sodium Sulfide – Dissolve about 15 gm of sodium sulfide crystals and 0.2 gm of sodium hydroxide into approximately 900 mL of DIW. Dilute to 1000 mL. This solution should be calibrated with the standardized sodium thiosulfate and iodine solutions before each use.

4. Quality Control

4.1 Quality Control

4.1.1 The minimum requirement for this method is an MDL study, and the analysis of laboratory reagent blanks, fortified blanks, duplicate sample analyses, and fortified matrix samples.

4.1.2 See Appendix I for QC Summary Table

4.2 Initial Demonstration of Performance

4.2.1 The initial demonstration of performance is used to characterize instrument performance, method detection limits, and linear calibration ranges.

4.2.2 Method detection limit (MDL) – The method detection will be established for each analyte, using DIW (blank) fortified at a concentration two to five times the estimated detection limit. To determine the MDL values, analyze at least seven replicate aliquots of the fortified reagent water and process though the entire analytical method. Perform all calculations and report the concentration in the appropriate units. Calculate the MDL as follows:

$$MDL = t * s$$

where:

t = Students' t value for 99% confidence level and a standard deviation estimate with n-1 degrees of freedom (t = 3.14 for seven replicates).

s = Standard deviation of the replicate analyses.

4.2.3 The MDL study for the AVS analysis will be determined gravimetrically.

4.2.4 The MDL study for the SEM analysis will be analyzed as outlined in Laucks SOP LTL-7105 (for ICP metals analysis by SW-846 method 6010) and LTL-7501 (for mercury cold vapor analysis by SW-846 method 7471).

4.3 Laboratory Reagent Blank (LRB)

4.3.1 Criteria

4.3.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the detection limit is reported. Method blank control limits are that contamination should not exceed half of the reporting limit.

4.3.1.2 The estimated reporting limits in μmoles/gm for AVS and SEM are listed in the table below:

Analyte	Reporting Limit
AVS	
Sulfide (S^{-2})	0.008
SEM	
Cadmium (Cd)	0.001
Copper (Cu)	0.002
Lead (Pb)	0.003
Mercury (Hg)	0.0001
Nickel (Ni)	0.006
Zinc (Zn)	0.005

4.3.2 Corrective action

4.3.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 20 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.4 Laboratory Fortified Sample Matrix (LFM)

4.4.1 Criteria

4.4.1.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting sample which are identified by the client as blanks. The LFM is analyzed exactly as a sample including exposure to all glassware, equipment, and reagents that are used with samples. The LFM is used to determine if sample matrix contributes to the bias of the analytical

results. The LFM is also known as a Matrix Spike. One matrix spike per 10 samples.

$$\% \text{ REC} = \frac{(R1 - S1) * 100}{TV}$$

where:

R1 = measured concentration in the spiked sample
S1 = measured concentration in the sample
TV= True Value concentration of analytes spiked in sample

4.4.1.2 The current %REC control limits for AVS and SEM are listed in the table below:

Analyte	% Recovery
AVS	
Sulfide (S^{-2})	50 – 150
SEM	
Cadmium (Cd)	75 – 125
Copper (Cu)	75 – 125
Lead (Pb)	75 – 125
Mercury (Hg)	75 – 125
Nickel (Ni)	75 – 125
Zinc (Zn)	75 – 125

4.4.2 Corrective action

4.4.2.1 If a trend in out of control %REC values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable recovery. Generally, if recoveries are out of control and no analytes of interest are detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

4.4.2.2 If both the matrix spike and blank spike are out of control low and there was no recoveries in the sample(s), then the sample results may be considered false negatives and re-analysis may be required or spike recovery limits may need to be re-evaluated.

4.5 Laboratory Fortified Blank (LFB)

4.5.1 Criteria

4.5.1.1 A method blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. The LFB is analyzed exactly as a sample including exposure to all glassware, equipment, and reagents that are used with samples. The LFB is used to determine if method analytes are recovering within the acceptable limits. The LFB is also known as a Blank Spike. One blank spike per 20 samples.

$$\% \text{ REC} = \frac{R2 * 100}{TV}$$

where:

R2 = measured concentration in the spiked blank
TV= True Value concentration of analytes spiked in blank

4.5.1.2 The current %REC control limits for AVS and SEM are listed in the table below:

Analyte	% Recovery
AVS	
Sulfide (S^{-2})	85 – 105
SEM	
Cadmium (Cd)	75 – 125
Copper (Cu)	75 – 125
Lead (Pb)	75 – 125
Mercury (Hg)	75 – 125
Nickel (Ni)	75 – 125
Zinc (Zn)	75 – 125

4.5.2 Corrective action

4.5.2.1 If a trend in out of control %REC values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable recovery. Generally, if recoveries are out of control and no analytes of interest are detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be

called for. Corrective actions should be discussed with the Quality Control Officer.

4.5.2.2 If both the matrix spike and blank spike are out of control low and there was no recoveries in the sample(s), then the sample results may be considered false negatives and re-analysis may be required or spike recovery limits may need to be re-evaluated.

4.6 Sample Duplicate

4.6.1 Criteria

4.6.1.1 At least one duplicate sample per 20 samples per matrix is required. RPD values are calculated as follows:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.6.1.2 The current % RPD control limits for AVS/SEM are listed in the table below:

Analyte	%RPD
AVS	
Sulfide (S^{-2})	30
SEM	
Cadmium (Cd)	20
Copper (Cu)	20
Lead (Pb)	20
Mercury (Hg)	20
Nickel (Ni)	20
Zinc (Zn)	20

4.6.2 Corrective action

4.6.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are out of control and no analytes of interest are detected in any of the samples, no immediate action will be taken on

that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation Procedures

5.1 Sample Preparation

5.1.1 Assemble glassware as illustrated in Appendix II.

5.1.2 Add a measured amount, approximately 10.0 gm, of wet sample to the reaction flask (first flask), magnetic stirring rod, and 100 mL of DIW. If the AVS concentration is expected to be high, less sample maybe used. It is not recommended to use less than 2.0 gm, due to sulfide oxidation and sample heterogeneity.

5.1.3 Add 25 mL of the 6M hydrochloric acid solution to the separatory funnel.

5.1.4 Fill the second flask with 175 – 200 mL of potassium acid phthalate solution. This solution is to buffer the hydrochloric acid from the reaction flask, as it would interact with the silver nitrate solution.

5.1.5 Fill the third and fourth flask with 175 – 200 mL of the silver nitrate solution.

5.1.6 Make sure that all of the rubber stoppers are on tight and start the magnetic stirrer.

5.2 Sample Analysis (AVS)

5.2.1 Purge the system for 10 minutes with 100 cc/min of nitrogen gas. Reduce the flow to 40 cc/min and continue purging for 10 minutes.

5.2.2 Begin to add the hydrochloric acid to the sample in the reaction flask. Do not drain all of the acid into the reaction flask, leave approximately 2 mL in the separatory funnel.

5.2.3 Reduce the flow of nitrogen to 20 cc/min and continue purging for 1 hour.

5.3 Sulfide Analysis

5.3.1 After the sample has been purged for one hour, check the fourth flask for precipitate. If there is precipitate in the fourth flask then the sample will need to be re-analyzed with less sample weight. If there is no precipitate in the fourth flask, then continue.

5.3.2 Filter the solutions from the third and fourth flask on to a preweighted 1.2 micron filter. Dry at 105°C, cool in a desiccators, and weigh.

5.3.4 Calculate the amount of silver sulfide (in gm) as the difference between the weight of dried silver sulfide with filter minus the weight of the preweighed filter.

5.4 AVS Calculations

5.4.1 Calculate the amount of sulfide in the sample (μmoles):

$$\text{Sulfide in Wet Sediment, } \mu\text{moles} = \frac{\text{Silver sulfide (gm)}}{247.8} * 10^6$$

5.4.2 Calculate the sulfide (AVS) concentration per gram of sample (μmoles/gm):

$$\text{AVS, } \mu\text{moles/gm DB} = \frac{S}{T * W}$$

Where,

S = Sulfide in Wet Sediment, μmoles
T = Total Solids (ratio of dry weight to wet weight)
W = Sample Weight (gm) as received

5.5 Simultaneously Extracted Metals Analysis (SEM)

5.5.1 After the generation of sulfide has been completed, filter the sediment suspension remaining in the reaction flask through a fast fiberglass filter (Whatman GF/D).

5.5.2 Transfer the solution to a 250 mL volumetric flask. Rinse the filtering flask 3 times with DIW and add the rinses to the volumetric flask. Dilute to volume with DIW.

5.5.3 Metals (cadmium, copper, nickel, lead, and zinc) will be analyzed by ICP as outlined in Laucks' SOP LTL-7105.

5.5.4 Mercury will be analyzed as outlined in Laucks' SOP LTL-7501.

5.6 SEM Calculations

5.6.1 Below are the calculations for the SEM portion of the analysis. "Metal" indicates only one of the target metals of interest and all of the metals of interest will need to be calculated.

5.6.2 Convert metal results from ug/L to μmoles/L:

$$X, \mu moles/L = \frac{U}{Y}$$

Where,

X = Metal result in µmoles/L
U = Metal result in ug/L
Y = Molecular weight of metal

5.6.3 Convert metal results from µmoles/L to µmoles:

$$Z, \mu moles = \frac{X}{V}$$

Where,

Z = Metal in Wet Sediment, µmoles
X = Metal result in µmoles/L
V = Final Volume in L

5.6.4 Convert metal results from µmoles to µmoles/gm DB and report value:

$$M, \mu moles/gm\ DB = \frac{Z}{T * W}$$

Where,

M = Metal in Dry Sediment, µmoles/gm
Z = Metal in Wet Sediment, µmoles
T = Total Solids (ratio of dry weight to wet weight)
W = Sample Weight as received

5.7 SEM to AVS Calculation

5.7.1 Calculate the ratio of SEM to AVS

$$SEM/AVS, \mu moles/gm\ DB = \frac{A}{B}$$

Where,

A = Is the sum of all the metals (cadmium, copper, lead, mercury, nickel, and zinc) results, µmoles/gm DB
B = The AVS result is determined in Section 5.4.2, µmoles/gm DB

5.7.2 A SEM:AVS ratio greater than one suggests that toxicity is possible while a ratio less than one suggests that the metals in the sediment are not toxic.

5.8 Bench Sheets

5.8.1 A copy of the bench sheets can be found Appendix III

5.9 Quality Control Reports

5.9.1 All results for quality control tests are entered into the lab database. If the data is for a data packet, copies of the printouts will be included in the data packet. Otherwise, their entry ID will be noted on the QC_DB Report Form. The routine minimum is a duplicate, if there is sufficient sample.

6. References

Draft Analytical Method for the Determination of Acid Volatile Sulfides in Sediment, EPA 821/R-91-100, December 1991

H.E. Allen and F. Gongmin, University of Delaware, Newark, Delaware

W. Boothman, Environmental Research Laboratory – Narragansett, U.S. Environmental Protection Agency, Narragansett, Rhode Island

D. Ditoro and J.D. Mahony, Bronx, New York

APPENDIX I

QC Summary Table

Laucks Testing Laboratories

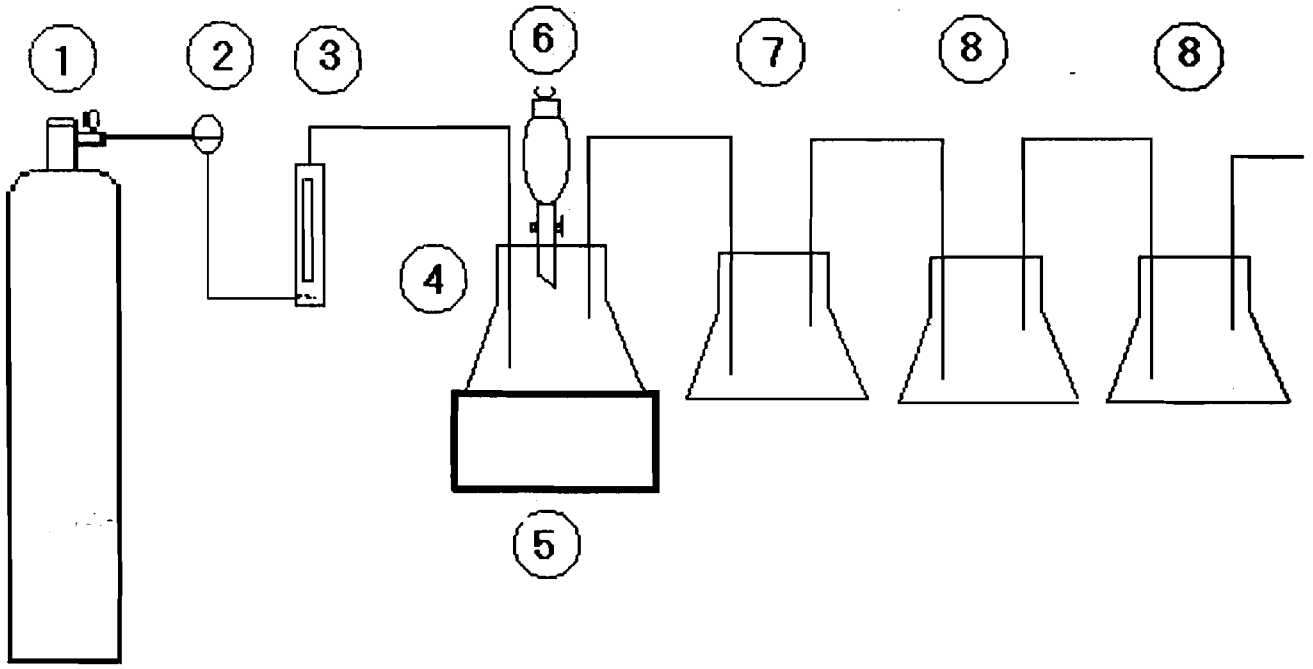
AVS/SEM QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank for AVS	<MDL	<½ of the Reporting Limit	1 per 20 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
Method Blank for SEM	<MDL	<½ of the Reporting Limit	1 per 20 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
Blank Spike for AVS % REC	85 – 105 %	85 – 105 %	1 per 20 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
Blank Spike for SEM % REC	None	75 – 125 %	1 per 20 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
MS for AVS % REC	None	50 – 150 %	1 per 10 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
MS for SEM % REC	None	75 – 125 %	1 per 10 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
Duplicate Analysis, for AVS % RPD	None	± 30	1 per 10 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate
Duplicate Analysis, for SEM % RPD	None	± 20	1 per 10 sample matrices	Re-analyze or use narrative comments	Corrective Action Form and/or narrate

APPENDIX II

Glassware Assembly

Glassware Assemblely



1 – Nitrogen Gas, 99.999%
2 – Regulator
3 – Flow Meter/Controller
4 – Reaction Flask
5 – Magnetic stirrer
6 – Separatory Funnel
7 – Potassium Acid Phthalate Solution Flask
8 – Silver Nitrate Solution Flasks

APPENDIX III

Bench Sheets

Laucks Testing Laboratories, Inc.

AVS Analysis Bench Sheet

Item	Date	Analyst	Checker
Prep #1	/ /		
Analysis	/ /		

Sulfide Standardization

Solution ID	Sulfide Spiked (L)	0.025 N Iodine (mL)	0.025N $Na_2S_2O_5$ (mL)	Sulfide Conc. (mg/L)	Average Sulfide Conc. (mg/L)
P. Blank	0.	10.			
aa					
bb					

AVS Bench sheet

Sample ID	Container ID	Sample Weight (gm)	Tare (gm)	Silver Sulfide + Tare (gm)	Silver Sulfide (gm)	Sulfide in Wet Sediment (µmoles)	AVS Result (µmoles/gm DB)

$$S, \mu mole = \frac{g}{247.8} * 10^6$$

Sulfide conc., mg/L = {[(a)(b) - (c)(d)] x 16.03} - e / f

$$AVS, \mu moles/gm\ DB = \frac{S}{T * W}$$

a = Normality of Iodine
b = Iodine (mL)
c = Normality of Sodium Thiosulfate
d = Sodium Thiosulfate (mL)
e = P. Blank Correction, mg = [(a)(b) - (c)(d)] x 16.03
f = Spiked Sulfide (L)

g = Silver Sulfide (gm)
S = Sulfide in Wet Sediment, µmole
T = Total Solids
W = Sample Weight (gm)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7010

Title: **Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy by SW846 Method 3010A**

Revision history:

Number	Date
1.	12/14/94
2.	06/03/96
3	2/1/01

Revised by: Stan Dittrick, Chemist — Date: 2/1/01

Reviewed by: Paul Swift, Inorganics Manager — Date: 02/01/01

Approved by: Harry Romberg, QA Officer — Date: 2-2-01

Approved by: Kathy Kreps, Laboratory Director — Date: 2-2-01

Controlled Document

No. 20 Assigned to: Tetra

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1. Introduction and Scope

1.1 This digestion procedure is used for the preparation of aqueous samples, TCLP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by inductively coupled argon plasma spectroscopy (ICP). The procedure is used to determine total metals.

1.2 Samples prepared by Method 3010A may be analyzed by ICP for the following:

Aluminum	Calcium	Magnesium	*Silver
*Antimony	Chromium	Manganese	Sodium
Arsenic	Cobalt	Molybdenum	Thallium
Barium	Copper	Nickel	*Tin
Beryllium	Iron	Potassium	Vanadium
Cadmium	Lead	Selenium	Zinc

* Although antimony, silver and tin are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3010A, Laucks has demonstrated adequate recovery for these analytes from this digestate.

1.3 This digestion procedure is not suitable for samples which will be analyzed by ICP-MS because hydrochloric acid can cause interference. Consult Method LTL-7008 for samples requiring ICP-MS analysis.

2. Summary of Method

2.1 A mixture of nitric acid and the material to be analyzed is refluxed in a covered beaker. This step is repeated with additional portions of nitric acid until the digestate is light in color or until its color has stabilized. After the digestate has been brought to a low volume, it is refluxed with hydrochloric acid and brought up to volume. If a sample goes to dryness, it must be discarded and prepared again.

3. Safety Procedures

3.1 These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

3.2 These procedures involve hot plates or hot blocks which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

3.3 Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before eating or going home for the day.

4. Equipment

- Plastic digestate bottles, precleaned
- 100-mL graduated cylinder, acid-washed
- Eppendorf or other micropipets
- Block digester at 90-95° C.
- Glass funnels, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, LTL-7003.

5. Reagents

5.1 Reagent Water. Reagent water will be interference-free. All references to water in the method refer to reagent water unless otherwise specified.

5.2 Nitric acid (concentrated), HNO_3, Trace Metals analyzed

5.3 Hydrochloric acid (concentrated), HCl, Trace Metals analyzed.

The method blank, prepared along with the samples, is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

6. Acid Digestion Procedure

See Appendix A for digestion log.
See Appendix B for flowchart.
See Appendix C for Quality Control solutions.
See Appendix D for Procedure Checklist
See Appendix E for Page from Standards Logbook

6.1 Transfer a 100-mL representative aliquot of the well-mixed sample to a digestate bottle and add 3 mL of concentrated HNO_3. If only one graduated cylinder is used for all

samples, the preparation blank is to be the last sample dispensed to assure the absence of sample carry-over. Place the digestate bottle on the block digester and carefully evaporate to 5 mL, making certain that the sample does not boil and that no portion of the bottom of the bottle is allowed to go dry. Make several holes in the top of one digestate bottle and suspend a thermometer in the lid. Place this digestate bottle full of water on the block digester. Make sure the thermometer does not touch the bottom. Record the temperature achieved during the digest on the digest log. Cool the digestate and add another 3-mL aliquot of HNO_3. Return the digestate bottle to the block digester and adjust the temperature, if necessary, so that a gentle reflux action occurs.

6.2 Continue heating, adding additional HNO_3 as necessary until the digestion is complete, indicated when the digestate is light in color or does not change in appearance upon additional heating. Evaporate the samples to 3 mL, not allowing any portion of the bottom of the beaker to go dry. After allowing the beakers to cool, add 5 mL of concentrated HCl and 5 mL reagent water. Reflux the samples for an additional 15 minutes to dissolve any precipitate or residue resulting from evaporation.

Note: If a sample is allowed to go to dryness at any stage of the digestion procedure, low recoveries will result. The sample must be discarded and prepared again.

6.3 After cooling the digestate bottles, add 5mL concentrated HCl to obtain a final acid concentration of approximately 10%. Bring the digestate to a final volume of 100 mL. If silicates or other insoluble material that could clog the nebulizer are present in the samples, the samples should be allowed to settle overnight prior to analysis. Filtration should be performed only if there is concern that the insoluble material will not settle out of solution. Place a funnel over the digestate funnel and use a Whatman no. 41 filter. Pour the sample in the filter. Acid wash filter apparatus to avoid sample contamination. Dilute the samples to a final volume of 100 mL. Samples should be stored in digestate bottles.

6.4 The prepared samples are then transferred to the metals analysis department. The digestion log is to reflect the time at the start and finish of the digest. In order to maintain strict chain of custody, the time and date when the digestates are relinquished to the analysis department, as well as the initials of the analyst accepting the digest, are recorded on the digestion log.

7. Quality Control

7.1 In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of 100 mL of reagent water and the appropriate amounts of nitric and hydrochloric acid, digested in exactly the same way as the samples. If only one graduated cylinder is used for all samples, the preparation blank is to be the last sample dispensed to assure the absence of sample carry-over. The blank should be labeled on

the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B111794ICPW01.

7.2 Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike / sample duplicate (MS/Dup) or matrix spike / matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. Two will be digested exactly as any other sample. The third must be spiked with the appropriate spike solutions determined by the analyst, and then treated as any other sample. **Spike additions must be witnessed by a second individual and initialed on the bottom of the digest log.** All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked and all glassware and digestate bottles marked appropriately.

7.3 Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log. A sample page from the standards logbook is included in Appendix E.

7.4 In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP LCSW) must be digested in exactly the same manner as a sample. A Blank Spike consists of 100 mL of reagent water spiked at the levels determined by the analyst as outlined in Section 7.3.

8. References

8.1 USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3010A.

Appendix A, Sample LTL-7010 Digestion Logs

LAUCKS TESTING LABORATORIES, INC.		ICP DIGESTION LOG	Place a √ in the box next to the method performed.		
	Digest start : date/time	Digest finish : date/time	SW-846 3005A		ILM04.0-water
SDG #: B0E79	01-17-01	1-17-01	SW-846 3010A	✓	ILM04.0-soil
Analyst: DLS	9:20	15:10	SW-846 3050A		SW-846 3050B

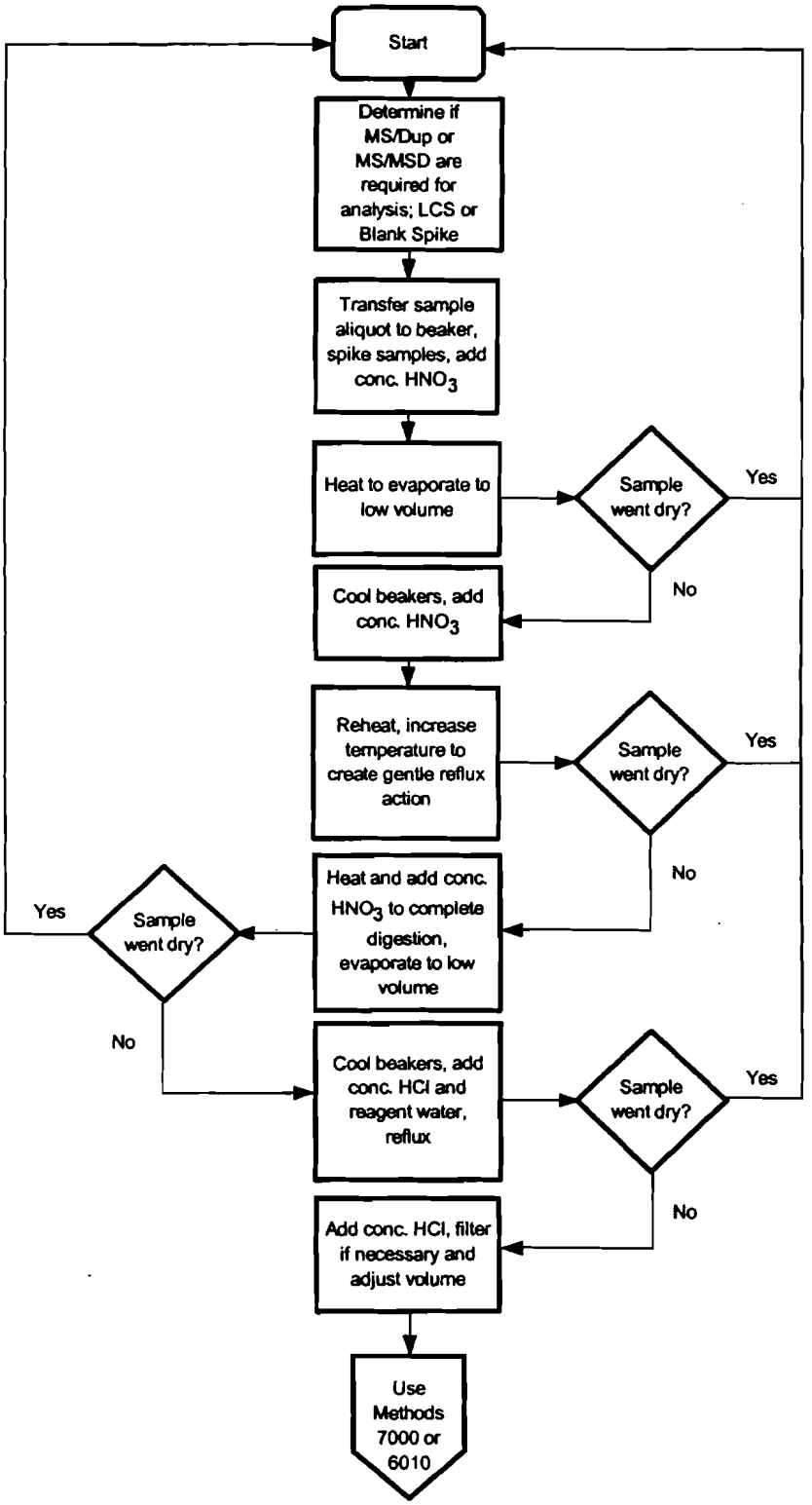
	Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color Bef	Color Aft	Clarity Bef	Clarity Aft	Text	Art	Comments	Bottle #
01	B0170171pw002	100	100	11	11	1	1				N/A
02	ICP LCS.01			11						ITE 13-11-8	1
03	010185-01			11							22
04	010185-02			9							23
05	-03			11							24
06	-04			1							25
07	-04D										25
☆ 08	-04S										25
09	-05										26
10	-06			1							27
11	-07	1	1	9	1	1	1				28
12											
13											
14											
15										01-17-01 DLS	
16											
17											
18											
19											
20											
21											
22											
23											
24											

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
	1-17-01 15:15 DLS	R 01/17/01		92°C

Reagents Lot Number	HNO_3	HCl	H_2O_2
	40161	40125	

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
P:\metals\bnchshee\icpdig.doc
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky) Artifacts: If "Yes", give description in the comments field.

Appendix B, Flowchart for LTL-7010


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or Blank Spike
Transfer sample aliquot to beaker, spike samples, add conc. HNO_3
Heat to evaporate to low volume
Sample went dry?
Yes
No
Cool beakers, add conc. HNO_3
Reheat, increase temperature to create gentle reflux action
Sample went dry?
Yes
No
Heat and add conc. HNO_3 to complete digestion, evaporate to low volume
Sample went dry?
Yes
No
Cool beakers, add conc. HCl and reagent water, reflux
Sample went dry?
Yes
No
Add conc. HCl, filter if necessary and adjust volume
Use Methods 7000 or 6010

Appendix C, Quality Control Solutions

ICP LCSW

The LCSW for the ICP digest was made by diluting 10 mLs of CLP-SPIKE and 50 mLs of LTL-6 solutions, and 2 mLs of conc. HNO_3 to 1000 mLs with type II water. A 100 mL aliquot of this solution was then digested.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Al	20000	Pb	500
Ba	20000	Mg	500000
Be	500	Mn	1500
Cd	500	Ni	4000
Ca	500000	K	500000
Cr	1000	Ag	1000
Co	5000	Na	500000
Cu	2500	V	5000
Fe	10000	Zn	2000
Sb	6000		

Other Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
As	1000	Tl	1000
Se	500		

Tables based on a final volume of 100 mL.

ICP Spikes

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with Type II water.

- Add 1000 µLs of CLP spike solution to water samples and 2000 µLs of CLP spike solution to soil samples prior to digestion.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500

Table based on a final volume of 100 mL for water digests, and 200 mL for soil digests.

ICP
CLP-LOW SPIKES

- Add the analytes from the two tables below into separate 100 mL volumetric flasks which contain ~50 mL of Type II water and 5 mL of HNO_3.

ICP-1

Analyte	Volume	Stock Concentration	Spike Concentration
Sb	500 µL	1000 ppm	5 ppm
Al	5000 µL	1000 ppm	50 ppm
Ba	2000 µL	1000 ppm	20 ppm
Be	100 µL	1000 ppm	1 ppm
Cr	500 µL	1000 ppm	5 ppm
Co	1000 µL	1000 ppm	10 ppm
Cu	500 µL	1000 ppm	5 ppm
Fe	2500 µL	1000 ppm	25 ppm
Mn	500 µL	1000 ppm	5 ppm
Ni	1000 µL	1000 ppm	10 ppm
V	1000 µL	1000 ppm	10 ppm
Zn	1000 µL	1000 ppm	10 ppm

ICP-2

Analyte	Volume	Stock Concentration	Spike Concentration
Ag	500 µL	1000 ppm	5 ppm

- Add 1000 µL of ICP-1 and 1000 µL of ICP-2 spike solutions to water samples prior to digestion. For soil samples, add 2000 µL of each spike solution prior to digestion. The final digest will contain:

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Sb	50 ppb	Al	500 ppb
Ba	200 ppb	Be	10 ppb
Cr	50 ppb	Co	100 ppb
Cu	50 ppb	Fe	250 ppb
Mn	50 ppb	Ni	100 ppb
V	100 ppb	Zn	100 ppb
Ag	50 ppb		

Table based on final volume of 100 mL for water digests, and 200 mL for soil digests:

PDCA
SPIKES and LCSW

The LCSW for the PDCA extraction was make by adding 10 mL SPEX ICV-1™ solution to 100 mL of Type II water prior to extraction. The spike was made by adding 1000 µL of CLP SPK solution to a 100 mL aliquot of the sample prior to extraction.

ICP Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Cd	50 ppb	Mn	500 ppb
Co	500 ppb	Ni	500 ppb
Cu	250 ppb	Ag	50 ppb
Fe	1000 ppb	V	500 ppb
Zn	500 ppb		

Graphite Furnace Analytes

Analyte	Concentration in Digest	Analyte	Concentration in Digest
Pb	500 ppb	Tl	2000 ppb
Se	2000 ppb		

Tables based on a final volume of 100 mL.

Appendix D, Procedure Checklist

SW846-3010A AQUEOUS DIGESTIONS (LTL-7010)

SWDIW*
ICP

QC: Blk, LCSW, Dup/Spk

- [ ] 100mL sample
- [ ] Add spikes
- [ ] 3mL HNO_3
- [ ] Reduce sample to 5 mL
- [ ] Cool 10 min
- [ ] Add 3 mL HNO_3
- [ ] Reduce sample to 3mL
- [ ] Cool 10 min
- [ ] Add 5 mL DIW and 5 mL HCl
- [ ] Heat 15 min
- [ ] Cool 10 min
- [ ] Add 5 mL HCl
- [ ] Final Volume 100mL

SWDFW
ICP-MS

QC: Blk, LCSW, Dup/Spk

- [ ] 100 mL sample
- [ ] Add spikes
- [ ] 0.5mL HNO_3
- [ ] 2 mL H_2O_2
- [ ] Reduce sample to 25-50mL
- [ ] Final Volume 100mL

* For SWDIW: If samples look clear use method SW846-3005A (LX_WM6).
For TCLP or dirty samples, use method SW846-3010A(SWDIW).

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Appendix E, Page from Standards Logbook

LAUCKS TESTING LABORATORIES, INC.
STANDARDS LOGBOOK

#	ANALYTE	ID or STOCK No.	STOCK CONC.	VOL/WT TAKEN	FINAL VOL.	FINAL CONC.	SOLVENT	PREP. DATE	INIT.	EXP. DATE	QC'D (Initials)
1	Hg Std	ME 12-98-2 11/1/01	998 µg/mL	100 µL	100 mL	1.0 ppm	0.15% HNO_3	11/7/01	EC	[illegible]	
2	Hg Std	ME12-98-2 11/1/01	998 µg/mL	100 µL	100 mL	1.0 ppm	0.15% HNO_3	11/20/00	EC	11/21/00	
3	ICP MS LCSW	ME12-98-05 11-1-01	1000 mL ~~500 mL 11/21/00~~	500 mL	1000 mL	various ~~various~~	2 mL HNO_3	11/21/00	DLH	11/1/2001	
4	Hg Std	ME12-98-2 11/01/01	998 µg/mL	100 µL	100 mL	1.0 ppm	0.15% HNO_3	11/21/00	EC	11/22/00	
5	ICP LCSW	ME 12-99-05 07/01/01	LTL-6 various	100 mL	2000 mL	various	2% HNO_3	11/22/00	DLH	07/01/01	
↓	↓	ME 13-4-3 9/01/01	clp spike various	20 mL	↓	↓	↓	↓	↓	09/01/01	
6	Hg Std	ME12-98-2 11/1/01	998 µg/mL	100 µL	100 mL	1.0 ppm	0.15% HNO_3	11/22/00	EC	11/23/00	
7	Hg Std	ME 12-98-2 11/1/01	998 µg/mL	100 µL	100 mL	1.0 ppm	0.15% HNO_3	11/27/00	EC	11/28/00	
8	CCV	QC Std 7 6/30/01	various MW12-57-10	2 ml	200 ml	various	5% HCl 1% HNO_3	11/27/00		6/30/01	
		QC Std 21 6/30/01	various	2 ml							
		SN 9/1/01	1000 µg/ml MW12-79-1	200 µl							
		stock std -	12-1/01 MW12-91-02	10 ml							

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-7501

Title: **Operation of the PE FIMS 400 for the Analysis of Hg for EPA Methods SW-846 7470/7471, 245.1/245.5, and CLP ILM04.1**

Revision history:

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1. Introduction and Scope

1.1 Method Description:

1.1.1 This SOP covers the cold vapor analysis of Hg using the Perkin-Elmer Flow Injection Mercury System, Model 400 (FIMS 400). Sample handling and preparation are as required by SW-846 methods 7470/7471 and 245.1/245.5. This SOP is also valid for the preparation and analysis of samples under CLP protocol when used in conjunction with the SOW ILM04.1. Where the requirements of these procedures differ, the more stringent requirements are used. The requirement of analyzing soil samples in triplicate under 7471 is not used except upon request.

1.1.2 After preparation and digestion to convert Hg to its ionic form, 0.5 mL of sample is introduced into a stream of carrier solution (3% HCl), mixed with a $SnCl_2$ suspension to reduce the Hg ion to its metallic form, which is then carried into an absorption tube through which passes light at a wavelength of 253.7 nm. The absorption is then measured.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are stored either in glass or plastic. The holding time is 28 days from collection. Soil samples are stored at 4° ±2° C. Water samples are stored at room temperature. Water samples are preserved by the addition of HNO_3 to a pH <2.

1.3 Interferences

1.3.1 This section covers interferences which may potentially occur with the FIMS associated with sample or standard preparation or gas or liquid flow through the system.

1.3.2 Foaming

1.3.2.1 If excess foaming results in liquid being transported into the quartz cell, the reagent stream should be stopped immediately, and the tubing and cell assemblies should be disassembled and cleaned.

1.3.3 Precipitates

1.3.3.1 All solutions used with the FIMS (samples, carrier solutions, and reducing agents) must be free of solid particles since they are transported through the narrow tubing and valve openings. In addition, the formation of precipitates during the course of a reaction must be avoided. When such a case occurs, an appropriate digestion or separation method should be used to remove the offending component.

1.3.4 Contamination

1.3.4.1 The detection limits obtainable using the FIAS-System are very often a function of the degree of contamination in the samples and the reagents rather than of instrument capabilities. Contamination is the main source of error when working at the nanogram levels typical for FIMS analyses. Careful operation of the instrument, selection of proper sample preparation techniques, and good laboratory cleanliness practices are the most important prerequisites for minimizing contamination.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.4.2 **LFB** - Laboratory Fortified Blank - A sample of DIW to which a known amount of Hg is added.

1.4.3 **CRA** – a sample of DIW to which a known amount of Hg is added. This is a low concentration to verify the curve is valid near the reporting limit of 0.2 µg/L.

1.4.4 **CCB** - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

1.4.5 **CCV** - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.4.6 **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.4.7 **Corr Coef, CC** - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a linear regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.4.8 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.4.9 **ICB** - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

1.4.10 **ICV** - Initial calibration verification - is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. This verification standard is from a source different from that used to make the calibration standards

1.4.11 **IDL** - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Determined quarterly using blanks to which a known quantity of standard has been added. The IDL is determined by analyzing 7 replicates spiked at 2-5 times the expected IDL on three non-consecutive days. The sum of the standard deviations of the three runs is the IDL.

1.4.12 **MDL** - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks annually.

1.4.13 **MDL** standard - Method detection limit standard - A standard prepared so that the concentration of Hg is 1-5 times the anticipated MDL

1.4.14 **LCS** - Laboratory Control Sample - A sample containing a known quantity of Hg used to verify digestion and analysis. LCSW indicates a water sample. It is used interchangeably with ICV. LCSS indicates a soil sample.

1.4.15 **RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.4.16 **PBW** - A blank carried through the sample preparation and digest procedures.

2. Equipment, Standards and Reagents

2.1 Equipment

2.1.1 Equivalent equipment may be used where appropriate.

FIMS 400 automatic Hg analyzer
Perkin Elmer AS-90 autosampler
Epson LQ300 dot-matrix printer
Hot water bath
BOD bottles
Analytical balance
Micropipettes: 20, 50, 100, 200, 500 and 1000 µl
50 mL and 15 mL plastic centrifuge tubes

2.2 Standards

2.2.1 All standards must be prepared in 0.15% HNO_3.

2.2.2 **1000 ppm Hg stock solution**: Vendor supplied. Currently purchased from Inorganic Ventures.

2.2.3 **Working Hg solution**: Dilute 100 µl of 1000 ppm Hg stock solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered in the standards logbook. This solution must be prepared daily. The identification number of the working standard must also be recorded on the bench sheet (digestion log-Appendix II).

2.2.4 **0.2 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 20 µl working Hg solution.

2.2.5 **0.5 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 50 µl working Hg solution.

2.2.6 **1.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 100µl working Hg solution.

2.2.7 **2.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 200 µl working Hg solution.

2.2.8 **5.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 500 µl working Hg solution.

2.2.9 **10.0 µg/L standard** - Place 100 mL DIW in a BOD bottle. Add 1000 µl working Hg solution.

2.2.10 **100 ppm Hg stock solution**: Vendor supplied. Currently purchased from Spex Certiprep.

2.2.11 **ICV Standard** - Dilute 1000µl of 100 ppm Hg stock solution to 100 mL in DIW to which 0.15 mL HNO_3 has been added. The identification number of the standard solution used and the dilution must be entered in the standards logbook and the digestion log (Appendix II). This solution must be prepared monthly from an independent stock solution, ie a different vendor than the stock solution used to prepare the calibration and quality control samples.

2.2.12 **ICV solution** - Add 100 mls DIW to a BOD bottle. Add sufficient ICV solution to produce a concentration of 1.0 to 5.0 µg/L. Currently a concentration of 4.0 µg/L is in use.

2.2.13 **CCV/CCB/Blank Spike (Soils only)** - In setting up the run, use the 5.0 µg/L standard for the CCV and Blank Spike; for the CCB use the calibration blank.

2.2.14 **MS/MSD or MS/Duplicate** - Add 500 µl of the working Hg solution to the samples being spiked. When performing Hg under CLP protocols use 100 µl of the working Hg solution and perform MS/Duplicate.

2.2.15 **LCSS** - Laboratory control soil sample. Concentrations and limits are certified by the vendor.

2.3 **Reagents**:

2.3.1 DIW - deionized water free of impurities.

2.3.2 H_2SO_4 (sulfuric acid) - concentrated, reagent grade

2.3.3 HNO_3 (nitric acid) - concentrated, reagent grade

2.3.4 HCl (hydrochloric acid) - concentrated, reagent grade

2.3.5 Stannous chloride - Add 27.5 g. $SnCl_2 \cdot 2H_2O$ (stannous chloride dihydrate) to 2.5 L DIW to which has been added 75 mL concentrated HCl.

2.3.6 $KMnO_4$ (potassium permanganate) - Dissolve 100 g. $KMnO_4$, marked "suitable for Hg determination", in 2 L DIW. This is a saturated solution. Sufficient undissolved $KMnO_4$ should be allowed remaining in the bottom of the bottle to assure a saturated solution.

2.3.7 $K_2S_2O_8$ (potassium persulfate) - Dissolve 50 g. $K_2S_2O_8$, marked "suitable for Hg determination", in 1 L DIW. This is a saturated solution. Sufficient undissolved $K_2S_2O_8$ should remain in the bottom of the bottle to assure a saturated solution.

2.3.8 $NaCl/NH_2OH{\cdot}HCl$ (sodium chloride/hydroxylamine hydrochloride) - Dissolve 240 g NaCl (table salt is generally used) and 240 g $NaCl/NH_2OH{\cdot}HCl$ (reagent grade marked "suitable for Hg determination") in 2 L DIW. Contamination has been experienced with this solution. For this reason a 50% solution should be analyzed by the FIMS 400 whenever a new bottle is made up. If the absorbance (peak height) exceeds .004 the solution of $NaCl/NH_2OH{\cdot}HCl$ should be disposed and remade.

2.3.9 Carrier solution - The carrier is a 3% HCl solution.

3. Safety Precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Because of the toxic nature of Hg vapor, inhalation or skin contact should be avoided.

3.1.6 The usual precautions should be taken in handling acids.

3.1.7 $SnCl_2$ is a skin and eye irritant; avoid contact.

3.1.8 The addition of hydroxylamine after digestion may release gas including chlorine. Avoid inhalation; use hood if necessary.

3.1.9 Waste disposal – See SOP LTL-2001 for general information on waste disposal. In addition, sample digestates are acidic in nature and must be neutralized prior to disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study (MDL).

4.1.1 The estimated MDL for waters is currently 0.05 µg/L. The estimated MDL for soils is currently 0.04 mg/kg.

4.1.2 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP on MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies for Hg water samples and soil samples are performed annually.

4.2 Initial Multi-Point Calibration:

4.2.1 Concentrations:

Standard Name	Concentration
S0	0.0 µg/L
S0.2	0.2 µg/L
S0.5	0.5 µg/L
S1.0	1.0 µg/L
S2.0	2.0 µg/L
S5.0	5.0 µg/L
S10.	10.0 µg/L

Standards are prepared fresh daily from the working standard in 0.15% HNO_3. See section 2.2.2 through 2.2.8 for the preparation details.

4.2.2 The calibration blank, the ICV, the LCS, the Blank Spike, the ICB and all standards are digested along with the samples being analyzed.

4.3 Calibration Criteria

4.3.1 Calibration curve – "best fit" of points in calibration curve. Linear correlation coefficient (R) must be equal to or greater than 0.995.

4.3.2 ICV - This is run immediately following a successful curve. This is to verify from a source of mercury independent from the calibration source that the curve is valid. For CLP and EPA 245.1/245.5 analyses the recovery limits are 80-120 %, for SW-846 7470/7471 the limits are 90-110%. If recovery is outside of the control limits an immediate reanalysis is permitted. If still out of control the curve must be reanalyzed followed by another ICV otherwise continue with an ICB.

4.3.3 ICB – This is run immediately following a successful ICV. This is to verify that there is no contamination and that the curve is valid. Limitations presently in use are ±0.1 µg/L (1/2 of the reporting limit). If recovery is outside the limit an immediate reanalysis is permitted. If still out of control, a corrective action must follow and a reanalysis of the curve.

4.3.4 A PBW and an LFB are run immediately following the successful calibration. The limitations on the PBW are the same as the limitation on the ICB. The limitations on the LFB are as set forth in the laboratory QC database (80-120%).

4.3.5 If the above criteria are not met, recalibration must be performed.

4.4 Continuing Calibration:

4.4.1 A CCV followed immediately by a CCB must be run every 10 samples and after the last sample. The ICB, PBW and Blank Spike count as samples for this purpose.

4.4.2 Criteria - For CCV's the recovery limitations are 80% to 120%. For blanks the limitations are ±0.1 µg/L.

4.4.3 Corrective action - If a CCV or CCB is out of control, an immediate reanalysis is permitted. However if the reanalysis is out of control a reanalysis of all samples since the previous successful CCV and/or CCB must be performed.

4.5 Matrix Spike

4.5.1 A sample is chosen at random from the samples to be analyzed, and 500 µl of the working standard is added (100µl for CLP). The spike level is then 5.0 µg/L(1.0 µg/L for CLP). The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect

of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery,\%} = \frac{(SS - S)}{SA} * 100$$

where:
SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.5.2 The recovery criteria are listed in the quality control database and will change from time to time. Current acceptable recovery limits are 85-116% for SW-7470. All other methods are 75-125%.

4.5.3 Corrective action

4.5.3.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of Hg from the LCS and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.6 Matrix Spike Duplicate

4.6.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} * 100$$

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.6.2 RPD control limits are listed in the quality control database and will change from time to time. The currently acceptable RPD limit is 20%.

4.7 Sample Duplicate

4.7.1 Sample duplicates are required when CLP practices are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed.

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} * 100$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analyses

4.7.2 The RPD control limits listed in the quality control database and will change from time to time.

4.7.3 Corrective action

4.7.3.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Assurance Officer.

5. Operation Procedures

5.1 Digestion Set-up - BOD bottles - Soil or Water:

5.1.1 Digestion log (Appendix II): The digestion log should list the sample numbers, the date, the name of the analyst, the time placed in the hot water bath, the time when the temperature reached 95 ± 5° C, and the time removed from the hot water bath. One should be prepared for each run. An example is attached as Appendix II. The numbers of each BOD bottle must be placed in the leftmost column of this form to identify which sample is in each BOD bottle.

5.1.2 Preparation of standards:

5.1.2.1 Prepare a working standard daily by adding 100 µl of the 1000 mg/L stock standard to 100 mL of 0.15% HNO_3 in DIW, giving a concentration of 1.0 mg/L of Hg.

5.1.2.2 Place 100 mL DIW in each of 9 BOD bottles. Mark these BOD bottles with the concentration of each standard in µg/L, plus one marked "Blank", one marked "ICV", and one more marked ICB.

5.1.2.3 Add from the working standard the following amounts to the following bottles:

0 µg/L	0 µl
0.2 µg/L	20µl
0.5 µg/L	50 µl
1.0 µg/L	100 µl
2.0 µg/L	200 µl
5.0 µg/L	500 µl
10.0 µg/L	1000 µl

5.1.2.4 To the ICV BOD bottle add that amount of the ICV standard which will produce a concentration from 1.0 µg/L to 5.0 µg/L. Currently a contration of 4.0 µg/L is in use.

5.1.2.5 The standards, the ICV and the ICB must be digested along with the PBW, Blank Spike and samples.

5.2 Digestion - Water Samples

5.2.1 These procedures are for methods SW-846 7470 and EPA 245.1 unless noted otherwise.

5.2.2 Place 100 mL DIW in the PBW BOD bottle.

5.2.3 Place 100 mL DIW + 500 µl intermediate standard in the Blank Spike BOD bottle.

5.2.4 Place 100 mL sample in the BOD bottle designated for that sample, being sure to place 100 mL sample in the designated duplicate, MS and MSD BOD bottles for the sample involved. TCLP samples and spikes should be diluted 20 mL/100 mL (dilution factor of 5) in DIW. The TCLP spikes should be diluted 2 mL/100 mL (dilution factor of 50) in DIW.

5.2.5 Add the required quantity (See Sec. 4.5.1) of the working standard to the MS and MSD BOD bottles.

5.2.6 Add the following reagents to each BOD bottle (for all methods):

5 mL concentrated H_2SO_4
2.5 mL concentrated HNO_3
15 mL $KMnO_4$ solution
8 mL $K_2S_2O_8$ solution

5.2.7 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.2.8 Place all the BOD bottles, being careful to keep them in order, in the hot water bath in a hood, and record the time on the digest log. Monitor the temperature of the hot water bath and record the time when the temperature reaches 90°C. Continue to heat the water bath, keeping the bath at 95°C ± 5°C for two hours.

5.2.9 Remove the BOD bottles from the hot water bath and allow to cool.

5.2.10 Add 7 mL $NaCl/NH_2OH{\cdot}HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear on standing after a few minutes.

5.3 Digestion - Soil samples

5.3.1 These procedures are (for Methods SW-846 7471 and EPA 245.5 unless noted otherwise):

5.3.2 Place 50 mL DIW in the calibration BOD bottles. Add appropriate working standard to each.

5.3.3 Place approximately 0.5 g, or for certain CLP and SW-846 analyses, approximately 0.2 g of sample in each of the sample BOD bottles to which that sample applies. Record the exact weight on the digestion log.

5.3.4 Add 3.75 mL HCl , 1.25 mL HNO_3 to all the BOD bottles and 10 ml DIW to each sample BOD bottle.

5.3.5 Place all sample BOD bottles in a hot water bath in a hood that is at 95°C ± 5°C. Maintain the heat at this level for 10 minutes.

5.3.6 Cool for 20-30 minutes and add 40 mL DIW and 15 mL $KMnO_4$ to all sample BOD bottles (also add 8 mL $K_2S_2O_8$ for CLP). Mix.

5.3.7 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.3.8 Place all BOD bottles in a hot water bath that is at 95°C ± 5°C. Record the time on the digestion log. Maintain the hot water bath at this temperature for 30 minutes. Remove the BOD bottles and allow to cool.

5.3.9 Add 7 mL $HCl/NH_4NH_2OH{\cdot}HCl$ solution to all BOD bottles and mix to remove color.

5.3.10 Add 50 mL DIW to all BOD bottles. Allow to cool.

5.4 Analysis on FIMS 400

5.4.1 This is a programmed analysis. This is a 6 point curve with a blank correction. The development of the calibration curve, including the ICV and ICB, is automatic. If the calibration is unsuccessful, recalibration is attempted. The 5.0 µg/L standard is used for CCV's; the calibration blank for CCB's, both of which are programmed to be run every 10 samples. If a CCV or a CCB fails to meet quality control specifications, the system stops and all samples since the last preceding valid CCV or CCB are reanalyzed. A CCV and a CCB are also run after the last sample in the run.

5.5 Sample Information sheet.

5.5.1 Click the basic functions button on the screen which appears after activating the program.

5.5.2 Click the "Sample Information" button on the toolbar.

5.5.3 A blank sample information sheet appears. Previous sample information sheets may be obtained by activating the File - Open menu, designating "Sample Information" from the dialog which appears. The sample information sheet should be filled in to tell the computer to which auto-sampler locations to go.

5.5.4 Sample ID column - Some typing can be saved by double clicking on the column title. On the resulting form the job order may be typed in and the sample numbers will then be incremented by 1 for the autosampler locations indicated. The sample ID's will then be filled in. Sample: "0102094-0001..0002. .".

5.5.5 Sample weight column - For soil samples the weight of the samples should be filled in. At the top, the volume units should be filled in "mL", the weight units "g".

5.5.6 Sample units column - Typing may be saved by double clicking on the column title and indicating the units on the dialog box which appears, and indicating the AS locations

involved. In the case of water samples this will ordinarily by "µg/L ". For soil samples the button "weight:weight" should be clicked and the desired units specified in the dialog box selected. If the dialog box has been properly filled in, the machine is programmed to compute the proper units (e.g. mg/kg). It should be kept in mind that this is on an as received basis, not a dry basis.

5.5.7 User dilution column - Where a dilution is involved (e.g. TCLP analyses) the dilution factor should be entered. As in the sample weight and sample units columns, above, typing may be saved by double clicking the column title.

5.5.8 The autosampler tray being used at present is "Tray B".

Sampler Location	Size	Contents
1	50 mL tube	Calibration Blank
2	50 mL tube	0.2 µg/L standard
3	50 mL tube	0.5 µg/L standard
4	50 mL tube	1.0 µg/L standard
5	50 mL tube	2.0 µg/L standard
6	50 mL tube	5.0 µg/L standard
7	50 mL tube	10.0 µg/L standard
8	50 mL tube	ICV
9	15 mL tube	ICB
10	15 mL tube	LCSS/LCSW
11	15 mL tube	CRA
12	15 mL tubes	PBW
13-109	15 mL tubes	Samples

5.5.9 All tubes should be marked with their contents before being filled. Then the tubes are filled from the BOD bottles and placed in the autosampler locations designated either by the method or by the sample information sheet.

5.5.10 At the beginning of each day of analysis, the following steps are taken to prepare the instrument:

5.5.10.1 The flows of the carrier solution and the reductant solution (the stannous chloride solution) are measured by the methodology suggested in the Perkin-Elmer handbook "Setting Up and Performing Analyses", 2.4.2-2.4.4.

5.5.10.2 The rinse container (autosampler location 0) is rinsed and filled with 3% HCl solution. This is then analyzed using the location button in the analysis window of the automated analysis window, and the curve produced displayed through the curve button in the toolbar. The curve should be flat and less than 0.0004 absorbance units.

5.5.11 Analysis - The analysis is ordinarily started by clicking the "Analyze All" button in the Automated Analysis window. If only calibration is desired, the "Calibrate" button may be used. If samples are to be analyzed by an already existing calibration curve use the "Analyze Samples" button.

5.5.11.1 Once the analysis has been started, the autosampler places the sampling probe in the sample/calibration cup. The peristaltic pump begins pulling sample through the sample loop. After the line has been purged, the injection valve cycles and injects the sample into the carrier flow. This flow is then combined with the $SnCl_2$ and mixed. Next it goes to a liquid/vapor separator. The liquid goes to waste and the vapor is transported into the absorption cell of the spectrophotometer. All samples and standards are measured in duplicate.

5.5.11.2 Run log - A run log will be printed as the analysis progresses. The print program is designed to transmit the data to be printed to the printer one page at a time. Thus several autosampler locations will be analyzed before anything appears on the printer. If anything else is printed (e.g. the sample information sheet) while the automated analysis window is open, the computer must be rebooted, or a run log will not be printed.

5.5.11.3 Additional samples may be analyzed after the run designated on the sample information sheet by using the "Select Location" button on the "Analyze" page of the Automated Analysis window. The autosampler locations should be filled in the resulting dialog. The instrument will then analyze the samples indicated. This may be used to reanalyze the samples with questionable results, or to analyze diluted samples.

5.5.11.4 Dilution - Where analysis results are above the high standard (10.0 µg/L) an aliquot of the sample diluted with the calibration blank may be analyzed as set forth in 5.5.11.3 above.

6. Reports

6.1 Data Packet Organization

6.1.1 A report is prepared using the reformat procedure on the main menu. The reformatted design is entitled "Summary". It is transferred to a floppy disk, which is then imported to a spreadsheet using Microsoft Excel 2000. This must then be formatted into the form included in Appendix III.

6.1.2 The final data packet is to include the following:

1) The report (See Appendix III).
2) The quality control database Report Form
2) The digestion log (Appendix II).
4) The run log, if required.

6.1.3 All results for quality control tests are entered into the quality control database. A summary report of all data entered must be included in the data packet. The routine minimum is a method blank report, an MS/MSD or MS/duplicate report and a SRM/LCSW.

6.2 Data Qualifying Flags

Flag	Definition
U	The analyte of interest was not detected, to the limit of detection indicated.

6.2.1 Control charts

7. References

USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 7470/7471

USEPA, Determination of Mercury in Water by cold vapor atomic absorption spectrometry, USEPA 245.1/245.5, most recent version, Method 245.1/245.5

CLP Inorganics Statement of Work, ILMO4.1

Appendix I - QC Summary Table

Laucks Testing Laboratories
Method 245.1/245.57471/7470, & CLP ILM04.1 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	R > 0.995	R > 0.995 6-point curve + blank	once per batch	recalibrate	run log
Initial Calibration Verification	SW846 90-110% EPA 245 and CLP 80-120%	Per the method	Once per batch	recalibrate	run log, report
Initial Calibration Blank	Below MDL	Below ½ R.L.	one per batch	recalibrate	run log, report
Method Blank (PBW)	Below R.L.	Below ½ R.L.	One per batch	Consult QC officer. Flag data or reanalyze.	Quality control database
Continuing Calibration Verification	80-120 %	80-120 %	every 10 samples and after the last sample	rerun samples since last CCV	run log
Continuing Calibration Blank	Below MDL	Below ½ R.L.	every 10 samples and after the last sample	rerun samples since last CCB	run log
Matrix Spike Recovery	See method	See quality control database	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database
MS/MSD RPD	See method	See quality control database	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Duplicate % Difference	See method	See quality control database	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database
Standard Reference Material (LCS) Recovery	Soil Vendor's limits Water See ICV	Vendor's limits	Once per 20	Recalibrate	Run log

Appendix - II - Sample Digestion Log

insert log here

Appendix III - Example Report Form

Hg in Water by SW-7470
Digested 11/13/2000
Analyzed 11/14/2000

Sample	Time	Value		% Rcvry	RPD	Flag
Calib Blank	11:49:54		µg/L			
S0.2	11:52:45		µg/L			
S0.5	11:55:38		µg/L			
S1.0	11:58:33		µg/L			
S2.0	12:01:30		µg/L			
S5.0	12:04:26		µg/L			
S10.0	12:07:22		µg/L			
ICV1 ME-13-4-1=4	12:10:26	3.951	µg/L	99%		
ICB	12:13:21	0.021	µg/L			
LCSW1	12:16:10	3.964	µg/L	99%		
CRA	12:19:03	0.199	µg/L			
PBW	12:21:57	0.017	µg/L			
0011185-01	12:24:51	0.023	µg/L			
011185-02	12:27:45	0.021	µg/L			
011185-03	12:30:42	0.024	µg/L			
011185-04	12:33:37	0.015	µg/L			
011185-04D	12:36:31	0.018	µg/L			
011185-04MS Spk=5	12:39:21	5.076	µg/L	102%		
CCV1=5	12:42:14	5.143	µg/L	103%		
CCB1	12:45:08	0.026	µg/L			
011185-05	12:47:58	0.017	µg/L			
011185-06	12:50:49	0.019	µg/L			
011185-07	12:53:40	0.021	µg/L			
011185-08	12:56:32	0.016	µg/L			
011185-09	12:59:24	0.017	µg/L			
011185-10	13:02:17	0.013	µg/L			
011185-11	13:05:12	0.009	µg/L			
0011143-02	13:08:05	0.016	µg/L			
011143-02MS Spk=5	13:10:59	5.160	µg/L	103%		
CCV2=5	13:13:54	5.143	µg/L	103%		
CCB2	13:16:46	0.028	µg/L			
011143-02MSD	13:19:39	5.150	µg/L	103%	0%	
0011149-02	13:22:34	0.018	µg/L			
0011167-02	13:25:31	0.016	µg/L			
011167-03	13:28:27	0.017	µg/L			
0011168-02	13:31:21	0.029	µg/L			
011168-03	13:34:10	0.017	µg/L			
0011196-02	13:37:01	0.021	µg/L			
011196-03	13:39:51	0.023	µg/L			
0011198-02	13:42:42	0.021	µg/L			
CCV3=5	13:45:34	5.163	µg/L	103%		
CCB3	13:48:28	0.026	µg/L			

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-8019

Determination of Non-Halogenated Volatile Organics by SW 8015B

Revision history:

Number	Date
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2	04/22/98
3	12/09/98
4	04/04/02

Revised by: Stacy Schulze, Analyst Date: 5/8/02

Approved by: Harry Romberg, QA Officer Date: 5-8-02

Approved by: Kathy Kreps, Laboratory Director Date: 5/9/02

Controlled Document

No. 20 **Assigned to:** TeTr

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1. Introduction and Scope

1.1 Method Description

1.1.1 Method 8015B is used in the determination of nonhalogenated volatile organic compounds and semivolatile organic compounds by gas chromatography. 8015B may also be applicable for the analysis of petroleum hydrocarbons, including gasoline range organics (GROs) and diesel range organics (DROs). However, this SOP is written strictly for the determination of nonhalogenated volatile organic compounds in soil, sediment, aqueous, and other matrices. This method employs sample introduction via a purge and trap sample concentrator.

1.1.2 A tabulated list of Laucks' typical analytes and their respective CAS numbers is located in Appendix I. The actual target analyte list may vary, depending on project-specific requirements.

1.1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations

Detailed below are any deviations from the published version of method SW8015. All deviations are followed as the standard operating procedure by Laucks Testing Labs.

1.2.1 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that low level soils be collected in VOA vials and preserved with sodium bisulfate. This option is not currently available with our instrumentation; therefore, we follow the method as described in section 1.4.2.2 of this document.

1.2.2 Current instrumentation does not allow separation on both columns of acetonitrile from the solvent peak in high level soil samples. Therefore, the result cannot be confirmed and only results from low level analyses are reported.

1.3 Method Interferences/ Decision-making Process

In order to attain lab-wide consistency among staff members for decision-making processes with regard to laboratory anomalies, several common items have been addressed in this SOP. Any occurrences not covered in this SOP should be discussed with the supervisor prior to implementing a solution.

1.3.1 Any samples analyzed subsequent to a high level sample (which is defined as yielding one or more target analytes above the calibration range) should be thoroughly examined for potential carry-over of the same target analyte(s). Sample syringes are rinsed between samples, but contamination may still occur. Corrective action in the form of reanalysis for possible carry-over should be performed and documented in the narrative. Screening samples prior to analysis will also help prevent such contamination.

1.3.2 All sample chromatograms should be reviewed for analytes which may not show up on the sample quantitation report due to data system error or retention time shift. All peaks should be examined and evaluated based on the retention times and sample concentration in order to prevent reporting false negatives.

1.3.3 Samples may be contaminated by a single compound (such as a solvent or petroleum product) which is detectable within the analytes' retention times. The use of the dual detectors should enable the analyst to differentiate between the analyte list and stray compounds; however, alternative calibration, detection, or quantitation procedures may be necessary if the compound is a project concern.

1.3.4 Contamination by other volatile organic compounds during shipment and storage may occur. Trip blanks prepared from PFW (for water samples and non-field-preserved soils) or P&T grade methanol (for field-preserved soils) travel with sample vials throughout sampling and storage to check for such contamination. Storage blanks are also analyzed once a week to check conditions within storage refrigerators.

1.4 Sample Collection, Sample Storage, Holding Times

1.4.1 Waters

1.4.1.1 Samples are collected with zero headspace in 40 ml glass VOA vials containing 1:1 HCl preservative and sealed with Teflon-lined caps. After preservation to a pH of ≤2, all samples are stored at 4°C ± 2°C.

1.4.1.2 The holding time to analysis is 14 days from date of collection for acid-preserved samples and 7 days for non-preserved samples. The pH values for the water samples are measured at the time of sample preparation and are noted in the instrument logbook and the client report.

1.4.2 Soils

1.4.2.1 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that low level soils be collected in duplicate using 5 grams and/or 1 gram aliquots which are to

be weighed out in the field. The soil samples are to be collected in a 40 ml vial containing water, sodium bisulfate, and a stir bar. 0.2 grams of sodium bisulfate are added for each gram of soil. Therefore, when collecting 5 grams of sample, the VOA vial should contain 1 gram of sodium bisulfate. The sodium bisulfate is added in order to ensure a sample pH of ≤2. The vials used for low level soils should have the tarred weight recorded on the label and be sealed with septum-lined screw caps. This procedure is not currently available for 8015B analysis due to instrument limitations.

1.4.2.2 Due to the previous technique's unavailability for 8015B analysis, Laucks uses the other option, which is the use of the EnCore™ sampler, the Purge-and-Trap Sampler™, or a cut plastic syringe to transfer the sample to the soil vial. If the EnCore™ sampler is used in the field, the samples may be stored for up to 48 hours. However, samples collected in this device should be transferred to the soil sample vials (and preserved) as soon as possible or analyzed within 48 hours. When preservation is not an option and soils must be analyzed straight from the EnCore™ sampler, a 7-day holding time to analysis may be negotiated with the client. The holding time for all other soils is 14 days from the date of collection.

1.4.2.3 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that high level soils be collected by using one of the following options:

1.4.2.4 Option 1: Samples will be collected and preserved with methanol in the field. An aliquot of the methanol is then analyzed by method 5030B in the laboratory. The holding time for soil samples collected in this manner is 14 days from the date of collection.

1.4.2.5 Option 2: Samples will be collected using an EnCore™ type sampler or equivalent and extracted (preserved) at the laboratory. Samples collected in this fashion may be stored for 48 hours but should be preserved as soon as possible. Once preserved, the holding time for sample analysis is 14 days from the date of collection.

1.4.2.6 Option 3: Samples will be collected in 2- or 4-oz glass containers and sealed with Teflon-lined caps with zero headspace. Samples are then extracted (preserved) in the laboratory. The holding time for soil samples collected in this manner is 14 days from the date of collection.

1.5 Definition of Terms

This section defines terms and acronyms as they are used in this SOP. Not all of the terms are defined here since it is assumed that the user of this SOP already understands their general meaning.

Blank Spike	A background free matrix (PFW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time samples or sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard. A blank spike is analyzed at a rate of 5% per matrix per day.
Calibration Check Standard	A standard analyzed after an initial calibration to check the accuracy of that calibration. The solution must be from a different source than the one used for the initial calibration. This solution may be the blank spike or QC check standard. See also blank spike and QC check standard.
CCV	Continuing Calibration Verification. Also known as the calibration verification standard. This standard is analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration.
CF	Calibration Factor. The ratio of analyte instrument response to nanograms injected. This term is defined in SW 846.
Corr Coef, r^2	Correlation Coefficient. A measure of the "goodness of fit" of a set of data to a linear regression model. The closer the value is to 1, the higher the degree of confidence in the correlation. The correlation coefficient must be greater than 0.990 to be acceptable. (Specific projects may require a greater degree of confidence by using a correlation coefficient of 0.995.)
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	Instrument Blank. An instrument blank contains the method surrogates and is analyzed to monitor for carry over between samples or sample extracts.

ICV	Initial Calibration Verification. The ICV is used in the same capacity as a CCV to check for continuation of the calibration; however, this standard is also used to adjust the mid-point of the retention time window on extended QC periods. Calibration is set by the initial multi-point curve.
IDL	Instrument Detection Limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method Detection Limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
Method Blank	A background free matrix (PFW for water, clean sand for soils/sediments) has surrogate added. This aliquot is taken through the entire analytical process. The purpose of the method blank is to demonstrate that interferences from the analytical system, glassware, laboratory contamination, and reagents are within tolerance. A method blank is analyzed at a rate of 5% per matrix per day.
MS/MSD	Matrix Spike and Matrix Spike Duplicate. A known amount of the target analytes and surrogates are added to a sample at a rate of 5% per matrix (1 MS/MSD set per 20 samples per matrix).
PFW	Purge-Free Water. Deionized water that has either been purged with high purity nitrogen for 15 minutes at 10 psi or has been boiled for 45 minutes in an environment free of all purgeable organics, cooled, and sealed for later use.
PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit. The value used when reporting a non-detect. It may be administratively, empirically, or contractually set.

QC check standard	Quality Control check standard. Referred to in this SOP as a blank spike (BS). A QC check standard is a requirement of SW 846 Method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC period	Quality Control period. An analysis sequence initiated by the analysis of one or more standards, followed by samples or sample extracts, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RF	Response Factor. The ratio of nanograms injected to peak response. This term is defined in the same way in both the CLP contract and SW 846.
RSD or %RSD	Relative Standard Deviation or Percent Relative Standard Deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.
RT	Retention Time. The time (in minutes) at which a target analyte elutes from the GC column.
RT window	Retention Time window. The +/- value applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of samples, QC, and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

Gas Chromatograph:	Hewlett Packard, 5890 I or II employing a low-dead-volume interface from the sample concentrator to the GC injection port.
Data System:	EZChrom data acquisition system, revision 6.6, Target/NT data processing system, revision 4.0 capable of off-line data storage or equivalent.
Detectors:	Dual FID detectors from Hewlett Packard or equivalent.
Purge and trap:	Tekmar LSC 2 or equivalent.
Autosampler:	Dynatech PTA-30W/S or equivalent.
Trap:	Tenax/OV1 (Supelco G Trap). Other packings for the trap may be substituted if equivalent or improved performance can be demonstrated.
Columns:	1) 30m x 0.45mm x 3.0 film bonded phase fused silica capillary (DB-624). 2) 30m x 0.45mm x 3.0 film bonded phase fused silica capillary (DB-VRX).

2.2 Supplies and Materials

Syringes:	5 and 10 ml Luerlok syringes.
Microsyringes:	10 and 25 µl syringes with a 0.006 in. I.D. needle (Hamilton 702N or equivalent) in addition to 50, 100, 500 and 1000 µl gas-tight syringes.
Volumetric flask:	Class "A" type volumetric flasks of assorted volumes ranging from 10 to 100 ml.
Micro reaction vials:	1 and 2 ml micro reaction vials with Mininert valves for storing current standard solutions.
VOA vials:	20 ml VOA vial with Teflon-lined caps.

Compressed gases:	Hydrogen 99.995% (high purity) or better. Helium 99.995% (high purity) or better. Air (breathing air grade without trace hydrocarbons) or purified air from the Balston 75-80 Zero Air Generator.
Reagent water:	Reagent water (also known as purge-free water) is deionized water that has been boiled for 45 minutes in an environment free of purgeable organics.
Methanol:	B&J Purge and Trap quality.
Disposable pipettes:	1, 5, and 10 ml disposable pipettes.
Stainless steel spatula	
Analytical balance:	Capable of accurately weighing to the nearest 0.0001 g for standard preparation and a top loading balance capable of weighing to the nearest 0.01 g for samples.
Note:	All of the above equipment may be substituted with equivalent or better equipment.

2.3 Standards

2.3.1 Preparation of Standards

2.3.1.1 The standards are currently prepared from either neat chemicals or commercially supplied premixed solutions. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at the concentration that the manufacturer guarantees if they are certified by the manufacturer or by an independent source.

2.3.1.2 The Neat/Standard Logbook is used for creating a discrete entry for each standard. The information entered into the book documents the traceability of all standards that are used (i.e., source, lot number, purity, date made, and expiration date). In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number.

2.3.1.3 Quality control is maintained on all solutions by verifying the standard solutions prior to use. This verification is accomplished through analysis of the new solution against a current 5-point calibration. Analyte values within 85-115 percent of their nominal value are considered acceptable.

2.3.1.4 Spiking solutions for the blank spikes and MS/MSD analyses are prepared in the same manner as the calibration standards. They are also used to verify the integrity of working calibration standards and the accuracy of the calibration curve. The spiking solutions are produced from standards purchased from a different vendor than the one used to purchase calibration standards.

2.3.1.5 All standards and spikes are stored in the VOA freezer at -10°C or lower. The working standards and spikes are stored in Mininert capped vials for a time period not to exceed three months or the expiration date of the parent standard, whichever is sooner. Prepared stock standards are stored for six months or the expiration date of the parent standard, whichever is sooner. Commercially prepared stock standards are stored until the manufacturer's expiration date or one year from opening, whichever is sooner. Standards are removed from the freezer and allowed to warm to room temperature prior to use.

2.3.2 Preparation of Surrogate Standards

2.3.2.1 If starting with a neat material, a measured amount of the neat chemical is placed in a glass volumetric, diluted to volume with methanol, and then transferred to an appropriate storage vial. A common dilution is approximately 125 mg of neat material into 25 ml of methanol. This dilution yields an intermediate solution of 5000 ng/µl, which is stored in amber vials for a time period not to exceed six months or the manufacturer's expiration date, whichever is sooner. Currently, we purchase separate certified solutions from Restek at concentration levels of 10,000 ng/µl each; they expire one year from opening or the manufacturer's expiration date, whichever is sooner.

Surrogates	**Source***	**Concentration**
Bromofluorobenzene	Restek	10,000 ng/µl
Trifluorotoluene	Restek	10,000 ng/µl

* or equivalent.

2.3.2.2 For field preserved soil samples, each surrogate is individually diluted ten-fold for a resulting concentration of 1000 ng/µl. These standards are stored for six months or the expiration date of the parent solution, whichever is sooner.

2.3.2.3 Aliquots of the 10,000 ng/µl stock solutions are taken from the surrogate standards and then combined in a volumetric flask and diluted with methanol to yield a working level multi-component surrogate of 200 ng/µl (for laboratory soil extraction). This solution is stored in a Mininert capped vial for a time period not to exceed six months or the expiration date of the parent standard, whichever is sooner.

Surrogate	Initial Conc.	Aliquot	Vol.	Final Conc.
Bromofluorobenzene	10,000 ng/µl	200 µl	10 ml	200 ng/µl
Trifluorotoluene	10,000 ng/µl	200 µl	10 ml	200 ng/µl

2.3.2.4 An aliquot of the 200 ng/µl solution is taken and then diluted ten-fold with methanol in a volumetric flask to yield a working level multi-component surrogate of 20 ng/µl (for water and low-level soil analysis). This solution is stored in a Mininert capped vial for a time period not to exceed three months or the expiration date of the parent standard, whichever is sooner.

Surrogate	Initial Conc.	Aliquot	Vol.	Final Conc.
Bromofluorobenzene	200 ng/µl	1 ml	10 ml	20 ng/µl
Trifluorotoluene	200 ng/µl	1 ml	10 ml	20 ng/µl

2.3.2.5 Surrogate standards are kept in the VOA freezer at -10°C or lower, removed prior to use, and allowed to warm to room temperature. An aliquot of the working solution is injected into a 10 ml syringe containing PFW or sample. Below are the surrogate concentrations and the preparation for water and low-level soil analyses.

Surrogate	Initial Conc.	Aliquot	Vol.	Final Conc.
Bromofluorobenzene	20 ng/µl	10 µl	10 ml	20 ng/ml
Trifluorotoluene	20 ng/µl	10 µl	10 ml	20 ng/ml

2.3.3 Preparation of Matrix Spike Standards

2.3.3.1 Certified sets of solutions containing the target analytes are purchased premixed from a commercial supplier at concentrations of 2000 ng/µl (for isobutyl alcohol, propionitrile, and 1,4-dioxane) or 1000 ng/µl (for acetonitrile). These solutions are stored until the manufacturer's expiration date or one year from opening the ampule, whichever is sooner. The oxygenate compounds (TAME, ETBE, DIPE, and TBA) are currently purchased as neats and diluted appropriately. These solutions are stored until the manufacturer's expiration date or five years from opening the ampule, whichever is sooner.

Matrix Spike Solution	Source*	Concentration
Isobutyl alcohol	Accustandard	2000 ng/µl
Acetonitrile	Accustandard	1000 ng/µl
Propionitrile	Accustandard	2000 ng/µl
1,4-Dioxane	Supelco	2000 ng/µl
TAME	ChemService	neat
ETBE	ChemService	neat
DIPE	ChemService	neat
TBA	ChemService	neat

* or equivalent.

2.3.3.2 Aliquots of the premixed solutions are taken and then diluted with methanol in a volumetric flask to yield a working level multi-component spike of 100 ng/µl (for isobutyl alcohol, acetonitrile, and propionitrile) and a single component spike of 500 ng/µl (for 1,4-dioxane). Certified neat chemicals of TAME, ETBE, DIPE, and TBA are purchased from a commercial supplier. Stock solutions are prepared from the neat chemicals by diluting 0.20g of material with methanol in a 10 ml volumetric flask, yielding individual solutions with concentrations of 20,000 ng/µl. These solutions are then diluted and mixed in methanol to generate a multi-component working standard at concentrations of 10-50 ng/µl. The oxygenate intermediate solutions are stored for a period of time not to exceed six months or the expiration date of the parent standard, whichever is sooner. All working solutions are stored in Mininert capped vials for an extended period of time not to exceed three months or the expiration date of the parent standard, whichever is sooner.

Matrix Spike Solution	Initial Conc.	Aliquot	Vol.	Final Conc.
Isobutyl alcohol	2000 ng/µl	50 µl	1 ml	100 ng/µl
Acetonitrile	1000 ng/µl	100 µl	1 ml	100 ng/µl
Propionitrile	2000 ng/µl	50 µl	1 ml	100 ng/µl
1,4-Dioxane	2000 ng/µl	250 µl	1 ml	500 ng/µl
TAME	20,000 ng/µl	5 µl	10 ml	10 ng/µl
ETBE	20,000 ng/µl	5 µl	10 ml	10 ng/µl
DIPE	20,000 ng/µl	5 µl	10 ml	10 ng/µl
TBA	20,000 ng/µl	25 µl	10 ml	50 ng/µl

2.3.3.3 Spikes are kept in the VOA freezer at -10°C or lower, removed prior to use, and allowed to warm to room temperature. An aliquot of the working solution is injected into a 10

ml syringe containing PFW or sample and an appropriate amount of the surrogate solution. Below are the spike concentrations and the preparation for the blank spike and MS/MSD analyses.

Matrix Spike Solution	Initial Conc.	Aliquot	Vol.	Final Conc.
Isobutyl alcohol	100 ng/µl	10 µl	10 ml	100 ng/ml
Acetonitrile	100 ng/µl	10 µl	10 ml	100 ng/ml
Propionitrile	100 ng/µl	10 µl	10 ml	100 ng/ml
1,4-Dioxane	500 ng/µl	20 µl	10 ml	1000 ng/ml
TAME	10 ng/µl	10 µl	10 ml	10 ng/ml
ETBE	10 ng/µl	10 µl	10 ml	10 ng/ml
DIPE	10 ng/µl	10 µl	10 ml	10 ng/ml
TBA	50 ng/µl	10 µl	10 ml	50 ng/ml

2.3.4 Preparation of Initial Calibration Standards

2.3.4.1 Certified neat chemicals of acetonitrile, propionitrile, and isobutyl alcohol are purchased from a commercial supplier. Stock solutions are prepared from the neat chemicals by diluting 0.20g of material with methanol in a 10 ml volumetric flask, yielding individual solutions with concentrations of 20,000 ng/µl. The stock solution of 1,4-dioxane is purchased as a certified, premixed solution from a commercial supplier at 2000 ng/µl. The oxygenates (TAME, ETBE, DIPE, and TBA) are purchased in a certified multi-component solution from a commercial supplier. Purchased solutions are stored until the manufacturer's expiration date or one year from opening the ampule, whichever is sooner. If additional target analytes are requested, these analytes may be ordered separately, in solution or as a neat material, and diluted to appropriate working concentrations through the described method.

2.3.4.2 The individual solutions are then diluted and mixed in methanol to generate a multi-component working standard at a concentration of 100 ng/µl (for acetonitrile, propionitrile, and isobutyl alcohol) or diluted to generate a single-component working standard at a concentration of 500 ng/µl (for 1,4-dioxane). The premixed oxygenates are diluted with methanol to generate a multi-component working standard with concentrations of 10-50 ng/µl. All solutions are stored in Mininert capped vials for a period of time not to exceed three months or the expiration date of the parent standard, whichever is sooner. The creation of the working surrogate standard was described in section 2.3.2.

The working standards contain the following compounds in each solution:

Analytes	Working Solution
Isobutyl alcohol	Mix 1
Propionitrile	Mix 1
Acetonitrile	Mix 1
1,4-Dioxane	Mix 3
Tert-amyl-methyl ether (TAME)	Mix 5
Ethyl-tert-butyl ether (ETBE)	Mix 5
Diisopropyl ether (DIPE)	Mix 5
Tert-butyl alcohol (TBA)	Mix 5
Surrogates	
Trifluorotoluene	Mix 2
Bromofluorobenzene	Mix 2 and Mix 4

2.3.4.3 Standards are kept in the VOA freezer at -10°C or lower, removed prior to making up the initial calibration standards, and allowed to warm to room temperature before use.

2.3.4.4 The initial calibration standards range from 20 µg/l to 300 µg/l for the non-dioxane/non-oxygenate compounds, from 50 µg/l to 3000 µg/l for 1,4-dioxane, from 1 µg/l to 500 µg/l for oxygenates, and from 2 µg/l to 40 µg/l for surrogates. The standards are prepared by withdrawing aliquots of the working solutions and injecting them into a 10 ml syringe containing PFW and an appropriate amount of the surrogate solution. After the curve has been analyzed, a method blank and a blank spike are also analyzed. The blank spike is prepared from a source different from that used for the curve and must be within 15% of the true value in order to pass.

Working Solution Conc. (non-diox/non-oxy compounds)	Aliquot	Final Volume	Concentration
100 ng/µl	2 µl	10 ml	20 µg/l
100 ng/µl	4 µl	10 ml	40 µg/l
100 ng/µl	8 µl	10 ml	80 µg/l
100 ng/µl	10 µl	10 ml	100 µg/l
100 ng/µl	20 µl	10 ml	200 µg/l
100 ng/µl	30 µl	10 ml	300 µg/l

Working Solution Conc. (1,4-Dioxane)	Aliquot	Final Volume	Concentration
500 ng/µl	1 µl	10 ml	50 µg/l
500 ng/µl	2 µl	10 ml	100 µg/l
500 ng/µl	10 µl	10 ml	500 µg/l
500 ng/µl	20 µl	10 ml	1000 µg/l
500 ng/µl	40 µl	10 ml	2000 µg/l
500 ng/µl	60 µl	10 ml	3000 µg/l

Working Solution Conc. (Oxygenates)	Aliquot	Final Volume	Concentration
10-50 ng/µl	10 µl into 100ml	10 ml of solution	1-5 µg/l
10-50 ng/µl	2 µl	10 ml	2-10 µg/l
10-50 ng/µl	5 µl	10 ml	5-25 µg/l
10-50 ng/µl	10 µl	10 ml	10-50 µg/l
10-50 ng/µl	20 µl	10 ml	20-100 µg/l
10-50 ng/µl	50 µl	10 ml	50-250 µg/l
10-50 ng/µl	100 µl	10 ml	100-500 µg/l

Surrogates	Aliquot	Final Volume	Concentration
20 ng/µl	1 µl	10 ml	2 µg/l
20 ng/µl	2 µl	10 ml	4 µg/l
20 ng/µl	5 µl	10 ml	10 µg/l
20 ng/µl	10 µl	10 ml	20 µg/l
20 ng/µl	15 µl	10 ml	30 µg/l
20 ng/µl	20 µl	10 ml	40 µg/l
20 ng/µl	30 µl	10 ml	60 µg/l

2.3.5 Preparation of a Continuing Calibration Standard

The concentration of the continuing calibration standard is the mid-point of the initial calibration curve. Due to its volatility, this solution is not stored for any period of time. Occasionally, the concentration of this standard is varied to further check the calibration.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 Many analytes in this method are known or suspected carcinogens. Analysts should handle all neat materials, standards, and samples as if they were potentially hazardous substances. Respirators and fume hoods should be employed for extremely hazardous materials.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.2 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.3 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.4 Methanol should be stored in a solvent locker and kept away from sources of heat and sparks. Contact should be avoided.

3.2 Waste disposal

3.2.1 All expired standards are discarded in the solvent waste container located in the prep area under the fume hood.

3.2.2 All high level samples are either returned to the client for disposal or are disposed into the appropriate waste container.

3.2.3 Soil samples are dumped into a 55 gallon drum in the warehouse where an outside source disposes of the waste.

3.2.4 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Chromatographic Conditions

Initial GC Temperature:	40°C
Hold Time:	3.0 min.

Ramp Rate:	8°C/min.
Final Temperature:	220°C
Final Time:	2.5 min.
Carrier Gas Flow:	10 ml/min.
Hydrogen Flow:	38 ml/min
Makeup Flow:	20 ml/min
Air Flow:	270 ml/min

4.1.2 Purge & Trap Parameters

Purge Time:	11 min.
Desorb Time:	4 min.
Bake Time:	16 min.
Desorb Preheat Temperature:	160°C
Desorb Temperature:	160°C
Purge Flow:	40 ml/min.
Purge Temperature:	40°C
Bake Temperature:	180°C
Desorb Flow Rate:	10 ml/min.

Dynatech Autosampler

Selection	Selected	Choice	Comments
Program set up	Auto	Man	Select Auto (use the arrow)
Start Delay	0.0		
Cycle Time	0		May vary depending on the GC
Aux Output	0		
Last Water	0		NEVER select anything but 0
Last Soil	1-30		Set according to # of samples
Blank Last	No		
Flush Vol.	5 ml		
Purge Setup	Soil	Water	Select Soil (use the arrow)
Standard	No		
Flow Rate	38 cc		
Line Heat	110C		
Sample Heat	40C		
Pre-Heat	0.0 min.		
Water Vol.	0.0 ml		
Pre-purge	0.0 min.		
Purge Time	11 min.		
Flushes	0		

Soil Stir	No	Yes	Low-level soils are stirred
Desorb Time	4 min.		
Water Trap Vol.	0		
Purge Setup	Soil	Water	System is verifying setup
Program set up	Auto	Man	System is ready to start, AUTO

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP for MDL determinations. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. The current MDL ranges at the time of this writing are located in Appendix VIII.

4.2.2 The above procedure is used whenever a major change of equipment occurs or a new method is developed. MDLs are checked quarterly by analyzing a standard at ½ the reporting limit.

4.3 Method Validation

Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically 10-50 times the MDL. In the absence of criteria for the initial demonstration of proficiency, the limits of 70%- 130% can be used.

4.4 Method Reporting Limits

Method reporting limits are based on the low level calibration standard, in accordance with SW 846 (see Appendix VIII). The limits are current at the time of the updating of this document.

4.5 Retention Time Windows

4.5.1 Prior to the analysis of any samples, it is necessary to establish retention time windows for the method by analyzing standards for all target analytes over at least a 72-hour period. These standards should be intermixed with real sample extracts in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds, and compute the standard deviation for each set of retention times.

4.5.2 The retention time window half-width is set at +/-3 times the standard deviation as previously determined. This operation must be repeated whenever major equipment changes are made or whenever the chromatographic method is modified.

4.5.3 In some cases, particularly for narrow bore capillary column analyses, the calculated retention time window half widths may be an unrealistically small value or even zero. If that is the case, then substitute a retention time window of a close eluting, similar compound to develop a valid retention time window.

4.6 Initial Multi-Point Calibration

Analyze standard solutions using a minimum of 5 different concentration levels. The lowest concentration should define the reporting limit. The highest concentration should define the upper usable working range of the detector. Evaluation criteria are detailed in Section 6.1. Refer to Appendix III for a tabulated list of calibration standard concentrations.

4.7 Calibration Check Standard

4.7.1 After the instrument is calibrated, the accuracy of the calibration should be checked. A standard from a different source than the calibration standards is analyzed. The acceptance criteria are detailed in Section 6.

4.8 Continuing Calibration Verification (CCV)

4.8.1 Prior to sample analysis, a mid-range calibration standard is analyzed. The computed calibration factor (CF) must meet the criteria detailed in Section 6.2.

4.8.2 The midpoint of the retention time window is set to the retention time for each target analyte in the initial CCV standard of the QC period to be used for compound identification. The retention times for analytes in subsequent CCV standards must be within the retention time windows as set by the initial CCV of the QC period.

4.8.3 Calibration verification standards are analyzed at the frequency of no more than ten samples between standards. All analyses, whether they are instrument blanks, blank spikes, or dry purges, are considered samples. In addition, this standard must be the last analysis made in the analysis sequence. Evaluation criteria are detailed in Section 6.3.

4.9 Sample Analysis

The ICV criteria and method blank criteria must be met prior to analyzing samples.

4.9.1 Analysis sequence

4.9.1.1 See Appendix IV for a detailed sample analysis sequence.

4.9.2 Sample Preparation

4.9.2.1 The samples are removed from the VOA refrigerator and allowed to warm to room temperature.

4.9.3 Water Sample Preparation

4.9.3.1 A 10 ml aliquot of the water sample is transferred to a 10 ml Luerlock syringe, where 10 µl of the surrogate solution is added. The sample containing the surrogate solution is then transferred to a 25 ml fritted soil vial, loaded onto the autosampler, and analyzed by SW5030B.

4.9.3.2 The sample volume and pH are recorded in the instrument logbook. The remaining sample can be transferred into a 20 ml VOA vial with zero headspace and sealed with a Teflon-lined cap.

4.9.3.3 If the analyte concentration exceeds the upper calibration limit, the sample must be diluted and reanalyzed.

4.9.3.4 To dilute a sample, a smaller sample aliquot is analyzed with PFW replacing the remainder of the 10 ml volume.

4.9.4 Soil Sample Preparation

The sample consists of the entire contents of the sample container. Do not decant any of the supernatant liquids. All soil samples must be stirred gently before removing an aliquot.

4.9.4.1 Low Level Soils

4.9.4.1.1 1.0 g of soil is weighed in an appropriate purge vessel, or the entire contents from the EnCore sampler is used. A magnetic stir bar is added. The actual sample weight should be recorded to the nearest tenth of a gram. (For example, 0.95 and 1.05 g should be recorded as 1.0 g, not 1 g.)

4.9.4.1.2 10 ml of reagent water containing the surrogate solution are transferred to the vial containing the soil sample. Refer to the water preparation above for details on the surrogate amount.

4.9.4.1.3 If the analyte concentration exceeds the upper calibration limit, the sample must be diluted and reanalyzed.

4.9.4.1.4 To dilute a sample, analyze according to the high-level soil, laboratory preserved procedure.

4.9.4.2 High Level Soils

4.9.4.2.1 Field preserved samples:

4.9.4.2.2 The client is provided with a 4oz. weighed glass container along with a 25 ml aliquot of methanol that contains a field surrogate (bromofluorobenzene) at 0.8 ng/ul. The client weighs approximately 25 g of soil into the container and then adds the methanol with surrogate.

4.9.4.2.3 Upon receipt by the laboratory, the sample is warmed to room temperature, the weight is recorded, and then 20 ul of the laboratory surrogate (trifluorotoluene) is added to produce a concentration of 0.8 ng/ul. (Note: samples requiring analysis for 1,4-dioxane do not have trifluorotoluene added.) The methanol and soil are re-mixed and then allowed to settle. A 250 ul aliquot is transferred to 10 ml of PFW, but no additional surrogate is added.

4.9.4.2.4 If the analyte concentration exceeds the upper calibration limit, the sample must be diluted and reanalyzed.

4.9.4.2.5 To dilute a sample, a smaller aliquot of the sample/methanol extract is analyzed.

4.9.4.2.6 Laboratory preserved samples:

4.9.4.2.7 Weigh 5.0 g of soil (recorded to the nearest 0.1 g) into a 20 ml VOA vial (or the entire contents from the EnCore sampler); then, add 0.1 ml of surrogate solution and 9.9 ml of methanol to the vial. Shake the sample for 2 minutes, and let the suspended solids settle before use, preferably overnight.

4.9.4.2.8 Transfer 100 µl of the aliquot to 10 ml of PFW. Record the sample weight and the aliquot amount in the instrument logbook.

4.9.4.2.9 If the analyte concentration exceeds the upper calibration limit, the sample must be diluted and reanalyzed.

4.9.4.2.10 To dilute a sample, a smaller aliquot of the sample/methanol extract is analyzed.

4.9.5 The same GC conditions apply to soil samples as water samples.

4.9.6 Compound Identification

4.9.6.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its retention time window on a second column. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria detailed in Section 6 are strictly adhered to.

4.9.6.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, as referenced in Part 1.3.2 under Method Interferences/Decision-making Process, the retention times of analytes and surrogates may be outside their expected windows due to sample matrix effects. The analyst may decide to readjust the target analytes' retention time windows on an ad hoc basis based on such an observed shift. If this corrective action is taken, it must be fully documented in the case narrative notes.

4.9.7 Compound Quantification

Target compound concentrations are calculated using the external standard calibration equations.

4.9.7.1 Aqueous samples

As expressed in SW846:

$$Concentrat\ ion(\mu g / L) = \frac{A_s \times V_t \times D}{CF_{avg} \times V_i \times V_s}$$

where:

CF_{avg} = Average CF from the initial calibration.

V_t = Total volume of the concentrated extract (µl). For purge-and-trap analysis, V_t is not applicable and therefore, is set at 1.

A_s = Area or height of the peak for the analyte in the sample.

V_i = Volume of extract injected (µl). For low level purge and trap analysis, $V_i = 1$. For medium level purge and trap analysis, Vi has a value.

D = Dilution factor of extract: the final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if a high level sample was diluted 100 µl to 10,000 µl and subsequently diluted an additional 100 µl to 10,000 µl, the expression would be: (10,000/100) * (10,000/100) = 100 * 100 = 10,000. If no dilution was made, D = 1.

V_s = Volume of the aqueous sample purged (ml). If units of liters are used for this term, multiply the results by 1000.

4.9.7.2 Non-aqueous samples

The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Concentration(\mu g / kg) = \frac{A_s \times V_t \times D}{CF_{avg} \times V_i \times W_s}$$

where:

W_s = Dry weight of sample extracted or purged (g). To obtain the dry weight, the wet weight of the sample must be multiplied by percent total solids (TS) expressed as a decimal. Therefore,

$$W_s(dry) = W_s(wet) \times TS \quad \text{where} \quad TS = \frac{100 - \%\text{Moisture}}{100}$$

All other variables have the same definition as for aqueous samples. When a non-aqueous sample is extracted for purge and trap analysis, V_i = volume of methanol extract added to reagent water for analysis.

5. Reports

5.1 Data Packet Organization

See Appendix V for a checklist detailing data packet organization.

5.2 Quality Control Reports

5.2.3 All results for quality control tests are entered into the lab QC database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Sample Result Reports

5.4 Data Qualifying Flags

Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U: The analyte of interest was not detected, to the limit of detection indicated.

B: The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.

J: The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.

D: The value reported is derived from the analysis of a diluted sample or sample extract.

P: When a dual column GC technique is employed, this flag indicates that calculated results from the two columns differ by more than 25%. Generally, we report the higher value (unless matrix interference has elevated one of the results).

E: The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.

C: The target analyte's presence was confirmed by GC/MS.

6. Quality Control

Refer to Appendix VI for detailed QA and corrective action requirements.

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 Initial calibration data can be evaluated in one of two ways: %RSD of the calibration factors (CF) or correlation coefficient (r^2) of a linear regression analysis.

6.1.1.2 The distinctions between these methods are: the %RSD method assumes a linear response with the calibration curve passing through the origin; the linear regression method assumes a linear response with an unforced intercept.

6.1.1.3 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

6.1.1.4 For a set of data points, the %RSD is determined by calculating the average calibration factor (CF_{avg}) and the standard deviation(σ). The %RSD is equal to 100 times the standard deviation divided by CF_{avg}:

$$\%RSD = \frac{100\sigma}{CF_{avg}}$$

6.1.1.5 Currently, Laucks employs the use of calibration factors. The %RSD must be less than 20% for all compounds.

6.1.1.6 To verify contamination free reagents and apparatus, a method blank is analyzed after the curve.

6.1.1.7 To verify the curve, a blank spike prepared from a second source is analyzed after the curve. It must be within 15% of the true value in order to pass.

6.1.2 Corrective action

If the criteria are not met, the instrument must be recalibrated.

6.1.3 Documentation

6.1.3.1 Copies of the calibration standards and the calculated %RSDs are stored with the raw data. If the initial calibration is not used, this information is noted in the instrument logbook.

6.2 Calibration Check Standard

6.2.1 Criteria

6.2.1.1 The calibration check standard is produced from a source different from the initial calibration standards and must be between 85 and 115 percent recovery.

6.2.2 Corrective action

6.2.2.1 If the percent recoveries are not acceptable, corrective action in the form of reanalysis or recalibration and reanalysis must be performed.

6.2.3 Documentation

6.2.3.1 Copies of the calibration check standard results and percent recoveries are stored with the initial calibration

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 At the beginning of the QC period and after every ten injections, a CCV standard is analyzed. The CF for each compound is calculated and the percent difference is calculated as follows:

$$\%Difference = \frac{CF_v - CF_{avg}}{CF_{avg}} \times 100$$

where:

CF_v = CF from the analysis of the verification standard.
CF_{avg} = Average CF from the initial calibration.

Note: The %D results cannot exceed the detailed CCV criteria of +/-15%.

The retention times for all target analytes must fall within the RT windows.

6.3.2 Corrective action

6.3.2.1 Check calculations, examine the chromatogram, check the integration, analyze another CCV, or perform instrument maintenance. To validate the quantitation of target analytes in analytical samples, the samples must be bracketed by CCVs where the percent difference is within +/-15%.

6.3.2.2 If the average percent difference is less than 15%, then the CCV standard is considered to be acceptable. If one of the bracketed samples has a detectable amount of the analyte whose %D has exceeded the allowable limit, then just that sample needs to be re-analyzed.

6.3.3 Documentation

6.3.3.1 Copies of the continuing calibration standards and the calculated %Ds are stored with the raw data. If the continuing calibration standard cannot be used, this information is noted in the instrument logbook.

6.4 Method Blank

6.4.1 Criteria

6.4.1.1 A method blank is used to verify contamination free reagents and apparatus. A method blank is analyzed prior to sample analysis. Method blank control limits are ½ the reporting limits for all current analytes. If analysis of the method blank results in detection of an analyte between the reporting limit and ½ the reporting limit, the chromatography is examined, and the method blank may be used if either the analyte was not detected in the samples or the analyst finds that the higher result is due to an anomaly that will not affect sample analyses.

6.4.2 Corrective action

6.4.2.1 Corrective action in the form of reanalysis of the method blank prior to sample analysis is performed when target analytes are present above the reporting limit or surrogate recoveries are outside the control limits.

6.4.2.2 It is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, method blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated.

6.4.3 Documentation

6.4.3.1 Copies of all method blank analyses are stored with the sample analyses raw data. The results for the method blank analyses are reported electronically via the laboratory's LIMS database. If a method blank cannot be used, this information is noted in the instrument logbook.

6.5 Blank Spike or QC Check Sample (LCS)

6.5.1 Criteria

6.5.1.1 A blank spike is prepared by adding a known amount of analyte to an aliquot of PFW for aqueous sample analysis or a weighed amount of sand for soil/sediment analysis instead of an actual sample. A method blank with added analytes is a blank spike. An aqueous blank spike is the same as a QC check standard, and the same standard solution can be used as a calibration check standard. The recovery criteria can be found in Appendix VIII.

6.5.2 Corrective action

6.5.2.1 The blank spike is used to determine the method's accuracy without including any matrix problems. Sample reanalysis is triggered by an out of control blank spike.

6.5.3 Documentation

6.5.3.1 Copies of method blank spike analyses are stored with the sample analyses raw data. The results for the blank spike analyses are reported electronically via the laboratory's LIMS database. The analyte and surrogate recoveries are monitored in a control chart to look for trends in the recoveries. If data from the blank spike analysis cannot be used, this information is noted in the instrument logbook.

6.6 Matrix Spike/Matrix Spike Duplicate

6.6.1 Criteria

6.6.1.1 A sample is chosen at random from the samples to be analyzed at a rate of one in twenty (5%). Then, duplicate aliquots are prepared by adding aliquots of spiking solution prior to prep and/or analysis. The matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The duplicate fortified analysis is to evaluate the precision of the method by monitoring the relative percent difference (%RPD). Recovery and RPD criteria can be found in Appendix VIII.

6.6.1.2 The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

The relative percent difference is calculated as follows:

$$RPD = \frac{S1 - S2}{\frac{(S1 + S2)}{2}} \times 100$$

where:

S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.6.2 Corrective action

6.6.2.1 Samples with spike recoveries and/or RPD values outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation and/or reanalysis. This process should also include evaluation of the recovery of surrogate compounds in the MS sample and recovery of matrix spiking compounds from the blank spike analysis. In all cases, a narrative explanation of the condition is required to detail the corrective actions taken. If a trend in out-of-control recoveries or RPD values is observed, the method used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.6.3 Documentation

6.6.3.1 The raw data for the matrix spike and matrix spike duplicate analyses are stored with the sample analyses raw data. The results for the matrix spike analyses are reported electronically via the laboratory's LIMS database.

6.7 Surrogate Recovery

6.7.1 Criteria

6.7.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor potential sample processing errors and matrix effects. Recovery limits are 70 – 130%. Surrogate compound recoveries are calculated as follows:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:

S_m = Concentration of surrogate measured in sample.
S_a = Concentration of surrogate added.

6.7.2 Corrective Action

6.7.2.1 Check calculations for possible error. Reanalysis is required for surrogate recoveries that exceed the established control limits, unless matrix interference is observed or surrogates are diluted out.

6.7.2.2 Out-of-control surrogate recoveries in the method blank require that the method blank be reanalyzed. Sample analyses are not performed until all criteria for the method blank analysis are met. Sample and QC analyses require reanalysis for out-of-control surrogate recoveries. If the sample analyses demonstrate out-of-control surrogate recoveries due to matrix interference, this corrective action is discussed in the narrative.

6.7.3 Documentation

Copies of all surrogate recoveries are stored with the sample analyses raw data. The surrogate recoveries for all samples, blanks, and QC analyses are reported electronically via the laboratory's LIMS database.

7. References

SW 846 - Test Methods for Evaluating Solid Waste - Method 8000B "Determinative Chromatographic Separations", USEPA - Office of Solid Waste and Emergency, Revision 2, December 1996.

SW 846 - Test Methods for Evaluating Solid Waste - "Non-Halogenated Organics using GC/FID", Method 8015B, USEPA - Office of Solid Waste and Emergency Response, Revision 2, December 1996.

SW 846 - Test Methods for Evaluating Solid Waste - "Purge and Trap", Method 5030B, U.S. EPA, Revision 2, December, 1996.

SW 846 - Test Methods for Evaluating Solid Waste - "Purge and Trap", Method 5035, U.S. EPA, December, 1996.

SW 846 - Test Methods for Evaluating Solid Waste - "Organic Analytes", Chapter Four, U.S. EPA, December, 1996.

APPENDIX I

Target Analytes And Surrogates

Analytes	CAS Registry Number
Isobutyl alcohol	78-83-1
Propionitrile	107-12-0
Acetonitrile	75-05-8
1,4-Dioxane*	123-91-1
Tert-butyl alcohol (TBA)	75-65-0
Diisopropyl ether (DIPE)	108-20-3
Ethyl-tert-butyl ether (ETBE)	637-92-3
Tert-amyl-methyl ether (TAME)	994-05-8
Surrogates	
Trifluorotoluene*	98-08-8
Bromofluorobenzene	460-00-4

APPENDIX II

Analyte Elution Order

Column: DB-624	Column: DB-VRX
Acetonitrile	Acetonitrile
Propionitrile	Propionitrile
Isobutyl alcohol	Isobutyl alcohol
Trifluorotoluene*	Trifluorotoluene*
1,4-Dioxane*	1,4-Dioxane*
Bromofluorobenzene	Bromofluorobenzene

* These two compounds co-elute. Therefore, when 1,4-dioxane is requested as a target analyte, the surrogate trifluorotoluene is omitted from the analysis.

Column: DB-624	Column: DB-VRX
Tert-butyl alcohol (TBA)	Tert-butyl alcohol (TBA)
Diisopropyl ether (DIPE)	Diisopropyl ether (DIPE)
Ethyl-tert-butyl ether (ETBE)	Ethyl-tert-butyl ether (ETBE)
Tert-amyl-methyl ether (TAME)	Tert-amyl-methyl ether (TAME)
Trifluorotoluene	Trifluorotoluene
Bromofluorobenzene	Bromofluorobenzene

Appendix III

Initial Calibration Concentrations (µg/l)*

Compound	STD1	STD2	STD3	STD4	STD5	STD6
Acetonitrile	20	40	80	100	200	300
Propionitrile	20	40	80	100	200	300
Isobutyl alcohol	20	40	80	100	200	300
1,4-Dioxane†	50	100	500	1000	2000	3000
Trifluorotoluene†	1	2	5	10	15	20
Bromofluorobenzene	1	2	5	10	15	20

* At the time of this writing, these are the current calibration standards, however, they are subject to change.
† Since these two compounds co-elute, the 1,4-dioxane calibration curve is analyzed separately.

Compound	STD1	STD2	STD3	STD4	STD5	STD6	STD7
Tert-amyl-methyl ether (TAME)	1	2	5	10	20	50	100
Ethyl-tert-butyl ether (ETBE)	1	2	5	10	20	50	100
Diisopropyl ether (DIPE)	1	2	5	10	20	50	100
Tert-butyl alcohol (TBA)	5	10	25	50	100	250	500
Trifluorotoluene	2	4	10	20	30	40	60
Bromofluorobenzene	2	4	10	20	30	40	60

APPENDIX IV

Analysis Sequence

Injection	Sample
1	Std 1
2	Std 2
3	Std 3
4	Std 4
5	Std 5
6	Std 6
	Method Blank
	A total of 10 samples and/or blanks, duplicates, or matrix spike samples.
	CCV
	A total of 10 samples and/or blanks, duplicates, or matrix spike samples.
	CCV (closing?)

or

Injection	Sample
1	ICV
	Method Blank
	A total of 10 samples and/or blanks, duplicates, or matrix spike samples.
	CCV
	A total of 10 samples and/or blanks, duplicates, or matrix spike samples.
	CCV (closing?)

APPENDIX V

Data Packet Check List

I. CLIENT DELIVERABLE - IF REQUESTED

II. QC Summary

_____ Surrogate Recovery Summary Report
_____ Blank Spike Report
_____ MS/MSD Report
_____ Method Blank Summary Report

III. Sample Data

(In numerical order)

_____ Organic Analysis Data Sheet
_____ Sample Confirmation Form
_____ Chromatogram/ Quantitation Report Primary
_____ Chromatogram/ Quantitation Report Secondary

IV. Standard Data

(In chronological order)

_____ Linearity Report Primary/Secondary
_____ CCV Reports, %Difference Primary/Secondary

Linearity Standards:
_____ Chromatograms/ Quantitation Reports Primary
_____ Chromatograms/ Quantitation Reports Secondary
Continuing Calibration Standards:
_____ Chromatograms/ Quantitation Reports Primary
_____ Chromatograms/ Quantitation Reports Secondary
Other Standards Used to Support Sample Data (if required):
_____ i.e., MDL Reports

V. Raw QC Data

_____ Method Blank Report
_____ Confirmation Form
_____ Chromatogram/ Quantitation Report Primary
_____ Chromatogram/ Quantitation Report Secondary

_____ Blank Spike Report
_____ Confirmation Form
_____ Chromatogram/ Quantitation Report Primary
_____ Chromatogram/ Quantitation Report Secondary

_____ Matrix Spike Report
_____ Confirmation Form
_____ Chromatogram/ Quantitation Report Primary
_____ Chromatogram/ Quantitation Report Secondary

_____ Matrix Spike Duplicate Report
_____ Confirmation Form
_____ Chromatogram/ Quantitation Report Primary
_____ Chromatogram/ Quantitation Report Secondary

VI. Bench Sheets

_____ SDG summary
_____ Injection Log
_____ Misc. Work Sheets: soil extraction, %TS, calculations, HTVR
_____ Standards Log for all standards used with work

VII. Reject Data

DO NOT COPY DO NOT PAGINATE

Acquired data not used to support sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX VI

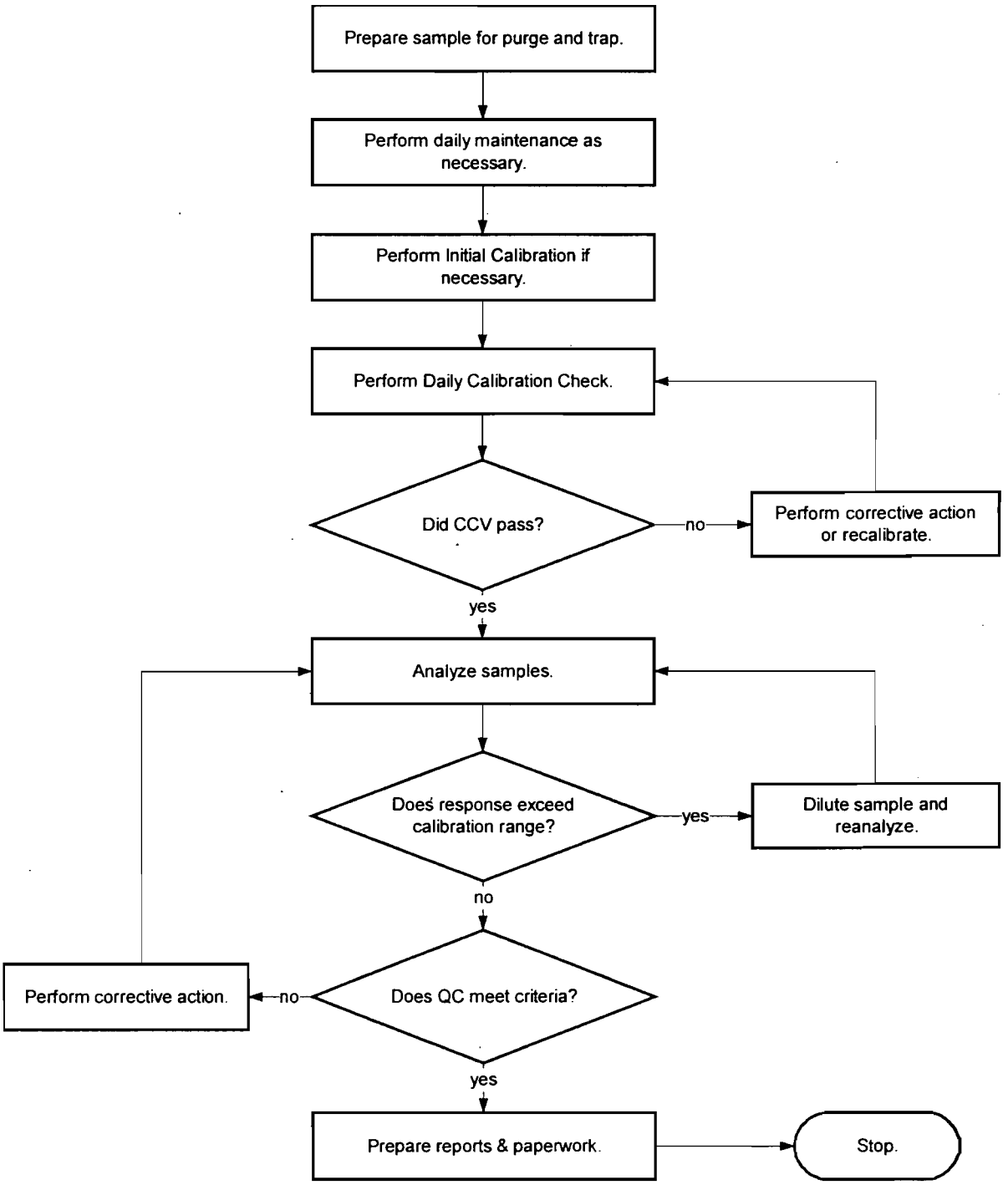
Method 8015B QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Holding Time	14 days from collection.	14 days from collection.	N/A	N/A	HTVR (holding time violation report) filed with data and noted in file and narrative.
Initial Calibration Curve	Minimum of 5 levels. If %RSD < 20, linearity assumed and average RF used. Alternatively, use linear regression to determine linearity.	Minimum of 5 levels. If %RSD < 20, linearity assumed and average RF used.	Initially and until ICV fails.	Reanalysis of out of control standards.	Copies of all raw data are filed.
Initial Calibration Verification	Mid-level calibration standard run every 10 samples. If not within ±15%, recalibrate.	Mid-level calibration standard run every 10 samples. If >15%D, reanalyze or recalibrate. RT are updated from this standard for all subsequent analyses.	At the beginning of every QC period.	Reanalysis of ICV in order to meet %D criterion. If reanalysis does not meet criterion, analyze initial calibration curve.	Copies of raw data filed and noted in instrument logbook.
Continuing Calibration Verification	Mid-level calibration standard run every 10 samples. If not within ±15%, recalibrate. Standard RT must fall within daily window or system is out of control.	Mid-level calibration standard run every 10 samples. If > 15%D, reanalyze for any out of control analyte which is detected in associated sample analysis. Standard RT must fall within daily window or system is out of control.	Every 10 samples.	Samples injected after criteria was exceeded must be reanalyzed.	Copies of raw data filed and notated in instrument logbook.
Internal Standards	Optional.	No internal standards are used at this time.			
Method Blank	One method blank per batch of up to 20 samples, or when there is a change in reagents, whichever is more frequent. Analysis of the method blank should demonstrate that interferences from the analytical system, glassware and reagents are under control.. Analytes present should be < MDL.	< 1/2RL for all analytes.	One method blank per batch of up to 10 samples.	Samples may not be analyzed until all criteria for the method blank are met.	Copies of raw data filed, and reported electronically. Daily control charts for all method blanks are recorded.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Surrogate Standards	Add surrogates to encompass range of temperature program. Results must fall within laboratory established control limits.	4-Bromofluorobenzene and/or trifluorotoluene. Percent recoveries must meet established laboratory control limits. See QC database for laboratory established control limits. (Currently 70 – 130%)	Surrogates are used in all standards, blanks, samples, and QC.	Reanalysis if surrogate recoveries exceed the established control limits. If reanalysis produces similar surrogate recoveries, then it may be due to matrix interference.	Any out of control surrogates are documented in associated file.
MS/MSD & Blank Spike	One blank spike per analytical batch. One MS/MSD per 20 samples or each batch of samples, whichever is more frequent.	Blank spike recoveries, must meet control limits established by laboratory. See control limits database for laboratory established limits.	One blank spike daily; MS/MSD per 20 samples.	Reanalysis of blank spike if out of control. If MS/MSD out of control and blank spike in control, no further action required.	MS/MSD recoveries are stored electronically. Raw data is stored with file. Out of control events are notated in file.
Sample Preservation and Storage	Water samples: pH ≤ 2 with HCL. All samples: store at 4° C.	Water samples: pH ≤ 2 with HCL. All samples: store at 4°C.	All water samples are to be preserved. Any samples to be stored at 4°C.	The pH is measured for all water samples. Client is notified if aqueous samples are not acid-preserved. pH cannot be determined until after analysis in order to maintain sample integrity.	pH of all water samples recorded in instrument logbook.
Standard Solution Expiration	Stock standards: 6 months. Store at 4° C with minimum headspace, away from light. Aqueous calibration standards: 1 hour , 24 hours if stored with zero headspace.	Stock standards: 6 months; Stored -10° to -20° C. Aqueous calibration standard: daily.			All standard data recorded in standard logbook.
Field Sample Amount Required	Water samples: 2 40 ml vials with zero headspace. Soils: glass, with Teflon-lines lids.	Water samples: 2 40 ml vial with zero headspace. Soil samples: 2 oz. glass containers, Teflon-lined lids.			Bottles received by laboratory documented electronically.
Applicability	Groundwaters, soils, sludges, water-miscible and non-water miscible wastes.				

APPENDIX VII

Flow Chart



APPENDIX VIII

MDL Ranges / Reporting and Control Limits

For Aqueous Samples:

			Blank Spikes		Matrix Spikes	
Compound	**MDL**	**RL**	**LCL - UCL**	**RPD**	**LCL - UCL**	**RPD**
Acetonitrile	3.660	40	65 - 133	30	57 - 126	20
Propionitrile	4.626	40	77 - 121	30	73 - 124	15
Isobutyl Alcohol	6.258	40	68 - 131	30	63 - 130	18
1,4-Dioxane	43.88	100	-		-	
a,a,a-Trifluorotoluene(s)	N/A		-	N/A	-	N/A
p-Bromofluorobenzene(s)	N/A		-	N/A	-	N/A

RL - Reporting Limit (ug/L) LCL - Lower control limit. UCL - Upper control limit. RPD - Relative percent difference.
MDL - Typical range for MDL (ug/L). (s) - Indicates a surrogate.
The reporting limit is based on a ten ml sample volume purged. The actual reporting limit will vary.

For Low Level Soil / Sediment Samples:

			Blank Spikes		Matrix Spikes	
Compound	**MDL**	**RL**	**LCL - UCL**	**RPD**	**LCL - UCL**	**RPD**
Acetonitrile	14.18	400	60 - 140	30	60 - 140	30
Propionitrile	13.19	400	60 - 140	30	60 - 140	30
Isobutyl Alcohol	13.17	400	60 - 140	30	60 - 140	30
1,4-Dioxane	38.55	1000	60 - 140	30	60 - 140	30
a,a,a-Trifluorotoluene(s)	N/A		-	N/A	-	N/A
p-Bromofluorobenzene(s)	N/A		-	N/A	-	N/A

RL - Reporting Limit (ug/Kg) LCL - Lower control limit. UCL - Upper control limit. RPD - Relative percent difference.
MDL - Typical range for MDL (ug/Kg). (s) - Indicates a surrogate.
The reporting limit is based on one gram of sample purged. The actual reporting limit will vary.

For High Level Soil / Sediment Samples (field preservation):

			Blank Spikes		Matrix Spikes	
Compound	**MDL**	**RL**	**LCL - UCL**	**RPD**	**LCL - UCL**	**RPD**
Acetonitrile	*	1600	60 - 140	30	60 - 140	30
Propionitrile	93.29	1600	60 - 140	30	60 - 140	30
Isobutyl Alcohol	67.26	1600	60 - 140	30	60 - 140	30
1,4-Dioxane	359.9	4000	60 - 140	30	60 - 140	30
a,a,a-Trifluorotoluene(s)	N/A		-	N/A	-	N/A
p-Bromofluorobenzene(s)	N/A		-	N/A	-	N/A

RL - Reporting Limit (ug/Kg) LCL - Lower control limit. UCL - Upper control limit. RPD - Relative percent difference.
MDL - Typical range for MDL (ug/Kg). (s) - Indicates a surrogate.
The reporting limit is based on purging a 250ul aliquot of 25grams of sample preserved with 25ml of methanol. The actual reporting limit will vary.
* Acetonitrile is visible only on the DB-624 column and cannot be confirmed when a large amount of methanol is present.

For High Level Soil / Sediment Samples (laboratory preservation):

			Blank Spikes		Matrix Spikes	
Compound	**MDL**	**RL**	**LCL - UCL**	**RPD**	**LCL - UCL**	**RPD**
Acetonitrile	*	8000	60 - 140	30	60 - 140	30
Propionitrile	631.7	8000	60 - 140	30	60 - 140	30
Isobutyl Alcohol	777.2	8000	60 - 140	30	60 - 140	30
1,4-Dioxane	2396	20000	60 - 140	30	60 - 140	30
a,a,a-Trifluorotoluene(s)	N/A		-	N/A	-	N/A
p-Bromofluorobenzene(s)	N/A		-	N/A	-	N/A

RL - Reporting Limit (ug/Kg) LCL - Lower control limit. UCL - Upper control limit. RPD - Relative percent difference.
MDL - Typical range for MDL (ug/Kg). (s) - Indicates a surrogate.
The reporting limit is based on purging a 100ul aliquot of 5grams of sample preserved with 10ml of methanol. The actual reporting limit will vary.
* Acetonitrile is visible only on the DB-624 column and cannot be confirmed when a large amount of methanol is present.

Note: At the time of this revision, analysis of the oxygenates is still under development, and MDL ranges have yet to be determined. MDLs are based on the low standard, which is currently 1 µg/l for TAME, ETBE, and DIPE; and 5 µg/l for TBA.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

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1 Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the procedures and specifications for instrumental analysis of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in water and soil following SW846 Methods 8081B and 8082A. Analysis is performed by gas chromatography using a single injection port and splitting into dual GC columns with electron-capture detectors. This system provides quantitation and confirmation of pesticides and Aroclors (PCBs) from a single injection. The following table lists the compounds that may be determined by these methods. Additional compounds, listed in Method 8081B, may also be determined.

SW8081B ROUTINE COMPOUNDS	
alpha-BHC	beta-BHC
delta-BHC	gamma-BHC (Lindane)
Heptachlor	Aldrin
Heptachlor epoxide	Endosulfan I
Dieldrin	4,4'-DDE
Endrin	Endosulfan II
4,4'-DDD	Endosulfan sulfate
4,4'-DDT	Methoxychlor
Endrin aldehyde	Endrin ketone
alpha-Chlordane	gamma-chlordane
Toxaphene	
SW8081B OPTIONAL COMPOUNDS	
**Chlordane (not otherwise specified)	Isodrin
Simazine	Atrazine

**When chlordane is requested, a technical chlordane multicomponent standard is analyzed and response factors are calculated for 3-5 major peaks (including the alpha- and gamma-isomers). If these peaks are present in the sample at similar ratios to those found in the technical chlordane standard, an average concentration is calculated from the peaks chosen for calibration. If the peaks are present in the sample at dissimilar ratios to those found in the technical chlordane standard, a calibration factor will be calculated for the standard based upon the sum of the responses for each of the peaks and the sample concentration will be calculated based on the sum of the same peaks in the sample.

SW8082A COMPOUNDS	
Aroclor-1016	Aroclor-1221
Aroclor-1232	Aroclor-1242
Aroclor-1248	Aroclor-1254
Aroclor-1260	

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.1.3 In some instances, samples being analyzed for PCBs will require sulfuric acid cleanup. In instances where samples are being analyzed for both pesticides and PCBs, an aliquot is separated prior to the sulfuric acid cleanup step, and is analyzed for pesticides only. The separation of aliquots prior to this cleanup will prevent the potential loss of target pesticide compounds.

1.2 Method Deviations

1.2.1 The analyte list is based on a combination of Method 8081B and 8082A analytes. Additional analytes are only added if requested by the client(s).

1.2.2 The continuing calibration standards' concentrations are varied per the Department of Defense (DOD) quality systems' requirement.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C±2°C. Water samples must be extracted within 7 days of sample collection, soil samples within 14 days of sample collection. All extracts must be analyzed with 40 days of sample preparation.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier — A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation.

Blank spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all HAZWRAP and NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration.
CF	Calibration factor. The ratio of analyte instrument response to nanograms injected. This term is defined in the same way in both the CLP contract and SW 846.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon-polishing filters, they are capable of providing organic-free water for use in method blanks and blank spikes.
IBLK	Instrument blank. This term is borrowed from CLP. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
MDL standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are in the range of 1x to 4x the empirically determined MDLs on an extract/digest basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set.
QC check standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC period	Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
Reporting Limit (RL)	The reporting limit is defined by the low level standard, also referred to as the practical quantitation limit (PQL).
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.
RT, Retention time	The time (in minutes) at which a target analyte elutes from a chromatography column.
RT window	Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.

Sequence	A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.
SRM	Standard Reference Material - A material containing known quantities of target analytes in a homogeneous matrix which approximates the matrix of the samples being analyzed. It is used to establish that the analytical process is in control.

2 Equipment List, Maintenance, and Standards

2.1 Chromatographic and Data System

2.1.1 This analysis requires a gas chromatograph with a programmable oven, heated injection port, dual electron-capture detectors, autosampler and an electronic data-acquisition system. Equipment list:
Gas chromatograph (Hewlett-Packard 5890, 5890A, 6890 w/µECDs)
Autosampler (Hewlett-Packard 7673A, 7683)
2 electron-capture detectors (Hewlett-Packard)
2 capillary chromatographic columns of dissimilar phase (J&W DB5 and DB608, 30m x 0.45mm megabore, J&W DB-XLB, DB35ms 30m x 0.32mm, or equivalents).
Helium carrier gas
5% methane/95% argon detector make-up gas,
EZChrom Data Acquisition System version 6.6, Target Analytical Software for NT Operating System version 4.02, Pentium PCs, or equivalent.

2.2 Maintenance

2.2.1 In order to prevent/reduce endrin breakdown, the following steps are used: 1) use clean and silanized Agilent liners, (splitless, single-taper liners for the 6890) 2) use only gold seals 3) reduce the injection port temperature e.g., from 250°C to 205°C 4) clean inlet with hexane and acetone 5) prime inlet with really high concentration standard 6) make sure syringe does not touch glass wool.

2.3 Standards

2.3.1 Commercially prepared, certified stock standards are used to prepare working solutions for all surrogates, calibration mixes, and spike mixes. The 2 calibration mixes (INDA and INDB) contain all of the individual pesticide analytes. The spike mix contains 3 representative individual pesticides and one Aroclor. These are ordered from Restek, Supelco or an equivalent supplier. Stock standards must be replaced by the manufacturers expiration date.

2.3.2 Calibration standards are prepared at 5 different concentration levels by dilution of the stock standards with hexane. High-level INDA and INDB mixtures are made at 16 times the PQL concentration. The lower levels are diluted by factors of 2, 4, 8 and 16. They must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.3.3 Multicomponent analyte calibration standards (Aroclors, chlordane, Toxaphene), with the exception of Aroclors 1016 and 1260, are prepared at one concentration level, by dilution of commercially purchased stock solutions in hexane. Standards of the Aroclors 1016 and 1260 are combined in a mixture and prepared at five concentration levels. These must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.3.4 A breakdown evaluation (EVAL) mix is prepared from dilution of stocks of 4,4'-DDT and endrin with hexane.

2.3.5 A surrogate mix is prepared from separate solutions which contain 2,4,5,6-tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB). These standards are made in hexane and added to all standard mixes (before dilution if applicable).

2.3.6 Refer to SOP LTL-1013 (Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials) for detailed instructions on standards preparation and storage.

2.3.7 Appendix I details the compounds and concentrations contained in all solutions.

3 Safety precautions and Waste Disposal

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they contain hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 The electron-capture detectors used in this analysis contain a radioactive source and should not be opened or otherwise tampered with.

3.2 Waste disposal

3.2.1 Solvent wastes generated in using this procedure (expired standards, old sample extracts, rinsates, etc.) should be emptied into the solvent waste container in the fume hood.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4 Calibration and Quality Control

4.1 QA Requirements and Corrective Action

4.1.1 Detailed in the following sections are applicable QA requirements and subsequent corrective actions to be applied to this analysis. A summary of these requirements can be found in Appendix IV.

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on MDLs, located in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method

validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.3.2 The precision of spike recoveries must meet or exceed the criteria tabulated in SW 846.

4.4 Retention Time Windows

4.4.1 Retention time window studies are conducted following the Laucks SOP on Establishing RTWs and the procedure detailed in SW 846, Method 8000B, which defines a window for each single and multi- component analyte based on the retention times taken from three standard injections over a 72 hour period.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made, whenever the chromatographic method is significantly modified, or whenever a column is replaced.

4.4.3 The calculated retention time window half widths are typically unrealistic values. Therefore RT windows have been administratively set at the values listed in OLM04.2 for megabore analyses (see table below) and at ±0.03 for capillary analyses. These values are typically wider than 3 times the standard deviation as determined above, but are more realistic.

Analyte	Megabore	Capillary
alpha-BHC	±0.05	±0.03
beta-BHC	±0.05	±0.03
gamma-BHC (Lindane)	±0.05	±0.03
delta-BHC	±0.05	±0.03
Heptachlor	±0.05	±0.03
Aldrin	±0.05	±0.03
alpha-Chlordane	±0.07	±0.03
gamma-Chlordane	±0.07	±0.03
Heptachlor epoxide	±0.07	±0.03
Dieldrin	±0.07	±0.03
Endrin	±0.07	±0.03
Endrin aldehyde	±0.07	±0.03
Endrin ketone	±0.07	±0.03
4,4'-DDD	±0.07	±0.03
4,4'-DDE	±0.07	±0.03
4,4'-DDT	±0.07	±0.03
Endosulfan I	±0.07	±0.03
Endosulfan II	±0.07	±0.03
Endosulfan sulfate	±0.07	±0.03
Methoxychlor	±0.07	±0.03
Tetrachloro-m-xylene	±0.07	±0.03
Decachlorobiphenyl	±0.10	±0.03
Aroclors	±0.07	±0.03
Toxaphene	±0.07	±0.03
Chlordane	±0.07	±0.03

4.5 Breakdown Evaluation (only required when analyzing for pesticides)

4.5.1 At the beginning of each analysis sequence a performance evaluation mixture (PEM) must also be analyzed. This is a separate solution containing 4,4'-DDT and endrin, and is examined for the breakdown products of these analytes (4,4'-DDD, 4,4'-DDE, endrin aldehyde, endrin ketone) which indicate the need for GC system maintenance. The percent breakdown must be less than 15% for both Endrin and 4,4'-DDT.

4.6 Initial Multi-Point Calibration for Pesticides and PCBs

4.6.1.1 Analyze single component pesticide standards using at least 5 different concentration levels for linear calibrations. The lowest concentration should be at a concentration near, but above, the method reporting limit or PQL. The highest concentration should define the upper usable working range of the detector. After the initial calibration is complete, an additional standard is analyzed. This additional standard is prepared from

a source that differs from the source of standards used in the initial calibration. This standard is performed in order to verify the initial calibration.

4.6.1.2 Analyze multi-component pesticide and PCB standard solutions at one concentration level, with the exception of the Aroclor 1016/1260 mixed standard. This standard should be analyzed at 5 different concentration levels for linear calibration. This step can be omitted if analyzing for pesticides only.

4.6.1.3 SW 846 allows the use of both linear and non-linear models for the calibration data. The option for non-linear calibration may be necessary to achieve low detection limits or to address specific instrumental techniques.

4.6.1.4 The choice of a specific calibration model should be made in one of two ways. The first is to begin with the simplest approach, the linear model through the origin, and progressing through the other options until the calibration acceptance criteria are met. The second approach is to use *a priori* knowledge of the detector response to choose the calibration model. Such knowledge may come from previous experience, knowledge of physics of the detector, or specific manufacturer's recommendations.

4.7 Linear Calibration

4.7.1 External standard initial calibration data is evaluated by determining the %RSD of the calibration factors.

4.7.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

4.7.3 The calculated CFs are tabulated and the %RSD calculated. There are no compound-specific criteria. All %RSDs must be within 20% for each analyte or averaged across all analytes in the calibration standard mix. Laucks uses 40% percent as the upper limit for all compounds as an internal guideline although it is not a requirement of SW 846.

4.7.4 Corrective action

4.7.4.1 If the criteria are not met, the instrument must be re-calibrated.

4.8 Non-linear Calibration

4.8.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed.

4.8.2 When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen

should have no more than four parameters, i.e., if the model is polynomial, it may be no more than third order, as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

4.8.3 The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches. A quadratic (second order) requires a minimum of six standards, and a third order polynomial requires seven standards. Do not force the line through the origin, i.e., do not set the intercept as 0, and do not include the origin (0,0) as a calibration point.

4.8.4 Whichever option is employed, a surrogate concentration must fall within the calibration range.

4.8.4.1 A set of three to five major peaks is selected for each multicomponent analyte. These should be characteristic of the multicomponent analyte in question. Retention time windows and calibration factors are generated for each of the peaks chosen.

4.8.5 Corrective action

4.8.5.1 If the criteria are not met, the instrument must be re-calibrated.

4.9 Initial Calibration Verification

4.9.1 Concentration and/or CF Criteria

4.9.1.1 At the beginning of an analysis sequence analyze a mid-range calibration standard. The computed calibration factor (CF) or concentration measurement must meet the criteria detailed below.

4.9.1.2 Using the appropriate calculation technique (average CF) compute either CFs or concentration values. For linear calibrations the ICV standard can be verified by calculating either the percent difference or the percent drift.

4.9.1.2.1 The percent difference calculation compares the ICV CFs to the mean CFs from the initial multi-point calibration. The percent difference is calculated as follows:

$$\%D = \frac{Cv - \overline{CF}}{\overline{CF}} \times 100$$

where:

C_v = Calibration Factor

$\overline{CF}$ = Mean Calibration Factor

4.9.1.2.2 The percent drift calculation compares the ICV calculated concentrations to the

theoretical (or actual) concentration of the ICV standard. The percent drift is calculated as follows:

$$\%D = \frac{C_c - C_T}{C_T} \times 100$$

where:

C_c= Calculated Concentration
C_T = Theoretical Concentration

4.9.1.3 There are no compound-specific criteria. The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.9.2 Corrective action

4.9.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.10 Updating Retention Time Windows

4.10.1 The retention time windows for compound identification are updated using the retention times for each target analyte in the mid-point calibration standard of the initial calibration as the center of the window and the previously determined retention time window half-width to establish the retention time range to be used for compound identification.

4.11 Instrument Blank

4.11.1 Criteria

4.11.1.1 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK or solvent rinse. This IBLK analysis is used only to make a judgment as to the possibility of carry-over into the sample extract immediately following the IBLK. Evaluation criteria are detailed below.

4.11.2 Corrective action

4.11.2.1 IBLKs used to monitor for possible carryover in high concentration extracts (those IBLKs optionally placed into the sequence following suspected high concentration extracts) are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be re-analyzed if there is a detectable amount of the analyte found in the IBLK.

4.12 Continuing Calibration Verification

4.12.1 The initial calibration is verified each 12 hours by injection of a calibration standard that alternates concentration levels. SW 846 requires that the calibration standard be injected

at a frequency of not less than once every 20 samples (the frequency of every 10 samples is recommended to minimize the number of reinjection due to QC failure). Laucks analyzes the continuing calibration standards every 10 samples or every 12 hours, whichever is deemed appropriate by the analyst. In addition, this standard must be the last injection made in the analysis sequence.

4.12.2 In accordance with DOD requirements, the concentration levels of the continuing calibration are varied approximately every 2 weeks when a new working solution is made. The concentration levels vary from the low level, mid-level and high level standards.

4.12.3 Criteria

4.12.4 After 12 hours, a CCV standard is analyzed. The CF or concentration for each analyte is calculated and the percent difference or percent drift is calculated as shown above.

4.12.5 The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.12.6 The retention times for all target analytes must fall within the RT windows established by the mid-point of the initial calibration.

4.12.7 Corrective Action

4.12.8 Check calculations or perform instrument maintenance. To validate the quantitation of target analytes in analytical samples, the samples must be bracketed by in-control CCVs. However, CCV CFs can be outside the control limits as long as this was due to an increase in response and the corresponding samples contain no detectable levels of the target analyte for which the CF is out of control.

4.13 Method Blanks

4.13.1 Criteria

4.13.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or one blank every 20 samples which ever is more frequent. Any analyte response above ½ the reporting limit is considered out of control.

4.13.2 Corrective action

4.13.2.1 Corrective action may necessitate re-extraction of the sample set. For example, if an analyte were found in the blank but not in any of the associated samples then the sample group may not require re-extraction. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or

elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.14 Blank Spikes

4.14.1 Criteria

4.14.1.1 A blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a blank spike. A blank spike is the same as a QC check standard. It is recommended that all single component pesticide target analytes of concern and a minimum of one Aroclor be spiked into the LCS. A subset of the single-component target analytes can be substituted for the full list if specified in project-specific contracts or workplans per the USACOE Shell document. In addition, the LCS and matrix spike should consist of the same components at the same concentration levels in order to accurately evaluate matrix interference in the MS/MSD samples.

4.14.1.2 Blank spike recoveries must meet the criteria specified in the quality control database.

4.14.2 Corrective action

4.14.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re- extraction and re-analysis would be triggered by an out of control blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

4.15 Matrix Spike

4.15.1 Criteria

4.15.1.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\%\text{Recovery} = \frac{MS - SA}{S} x100$$

where:

MS = concentration in spiked sample

SA = native concentration in unspiked sample
S = spike amount

4.15.1.2 The recovery criteria are detailed in the QC database. In the instance that the native target analyte concentration is greater than 5x the spike concentration, the MS recovery control limits do not apply. In this case, treat the MS/MSD pair as duplicates and report them as such in the quality control database.

4.15.2 Corrective action

4.15.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.16 Matrix Spike Duplicate

4.16.1 Criteria

4.16.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$\%RPD = \frac{|\chi - \delta|}{(\chi + \delta)/2} x100$$

where:
χ = measured concentration for MS sample
δ = measured concentration for MSD sample

4.16.1.2 RPD control limits are detailed in the QC database.

4.16.2 Corrective action

4.16.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

4.17 Surrogate Recovery

4.17.1 Criteria

4.17.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\% \text{ recovery} = \frac{S_m \times 100}{S_a}$$

where:

S_m = concentration of surrogate measured in extract
S_a = concentration of surrogate added

4.17.1.2 Detailed surrogate recovery control limits are tabulated in the QC database.

4.17.2 Corrective Action

4.17.2.1 Check calculations for possible error. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recovery since non-GC/MS methods cannot separate co-eluting interferences. Hence corrective action is not required for high surrogate recoveries.

4.17.2.2 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and re-analyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

5 Operation procedures

5.1 Chromatographic Conditions

5.1.1 The following general operating parameters are used on gas chromatographs to perform this method utilizing megabore (0.45 mm) columns:

Carrier gas:	helium
Column flow:	8 mL/min
Make-up gas:	argon/methane (95%/5% High Purity grade)
Make-up flow:	70 mL/min
Injector:	Grob-type, splitless
Injector temperature:	205°C
Injection:	splitless
Injection volume:	2µL (split - 1 µL per column)
Initial temperature:	150°C
Initial hold time:	0.5 min
Temperature ramp:	4°C per min
Final temperature:	275°C
Final hold time:	9 min
EC detector temperature:	350°C

5.1.2 The following general operating parameters are used on the 6890 gas chromatograph equipped with 0.32 mm columns:

Carrier gas:	helium
Make-up gas:	argon/methane (95%/5% High Purity grade)
Column flow:	2.6 mL/min
Make-up flow:	35 mL/min
Pulsed pressure:	55.0 psi
Pulsed time:	0.60 min.
Purge flow:	50.0 mL/min
Injector:	splitless, single-taper
Injector temperature:	250°C
Injection:	splitless
Injection volume:	1 µL (split – 0.5 µL per column)
Initial temperature:	150°C
Initial hold time:	0.5 min
Temperature ramp:	25°C per min
Final temperature 1:	250°C
Final time 1:	1 min
Rate 2	15°C per min
Final temperature 2:	340°C
Final time 2:	2.00 min
EC detector temperature:	350°C

These GC conditions should be optimized for analyte separation and sensitivity with a particular pair of columns and detectors. Once optimized, the same conditions must be used for the analysis of all standards, samples, blanks and spikes.

5.2 Sample Analysis

5.2.1 Analysis sequence

5.2.1.1 See Appendix II for a detailed analysis injection sequence.

5.2.2 Compound Identification

5.2.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the column 1. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Retention time windows are established as previously described and the absolute retention times are updated each QC period. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes.

5.2.2.3 Identification of multicomponent analytes occurs when the retention times and ratios of each of the multicomponent peaks present in the sample match the peaks chosen for the particular multicomponent analyte in the initial calibration. These same conditions must be met on both columns in order to confirm the presence of the multicomponent analyte. The experienced analyst's judgment weighs heavily in evaluating the patterns of multicomponent analytes with regards to weathering and matrix interferences.

5.2.3 Compound Quantitation

Target compound concentrations are calculated using the following equations:

5.2.3.1 Aqueous samples

5.2.3.1.1 The external standard equation, as expressed in SW 846 is:

$$\text{Concentration } (\mu g / L) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(V_s)}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
CF_m = Multi-point average CF
V_i = Volume of extract injected, µL,
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was diluted 100 µL to 1000 µL and subsequently diluted an additional 100 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t = Volume of total extract, µL.
V_s = Initial sample size, mL.

The reported concentration for multicomponent analytes calculated is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.3.1.2 To report concentrations in alternate units, apply an appropriate factor:

$$mg/L = \mu g/L * 0.001$$

5.2.3.2 Non-aqueous samples

5.2.3.2.1 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Conc.(\mu g / kg) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(W)(TS/100)}$$

where:
W = Weight of sample extracted or purged, grams.
TS = Total solids, percent.

The reported concentration for multicomponent analytes is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.4 Sample Dilution

5.2.4.1 If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. The dilution should be made so that the concentration of the analyte requiring dilution is in the mid to upper calibration range.

6 Reports

6.1 Data Packet Organization

6.1.1 See Appendix III for a checklist detailing data packet organization.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the quality control database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and an MS/MSD report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the reporting limit indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
P	When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. If the results from one column is significantly higher (25%) the chromatogram is checked for overlapping peaks, or irregular baseline integration. If no anomalies are discovered, the higher result is reported in order to employ the conservative approach relative to protection of the environment.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
C	The target analyte's presence was confirmed by GC/MS.

6.4 Control Chart(s)

6.4.1.1 The recovery values for gamma-BHC, Heptachlor, Aldrin, Aroclor 1260, TCMX, and DCB in the Blank Spike are plotted on control charts.

7 References

1). U.S. EPA Contract Laboratory Program Contract, Exhibit D, Section III, March 1990

2). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method 8000B, Gas Chromatography, Revision 2, December 1996.

3). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Organochlorine Pesticides by Gas Chromatography, Method 8081B, Revision 2A, May 1999 .

4). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Polychlorinated Biphenyls, (PCBs) by Gas Chromatography, Method 8082A, Revision 1A, May 1999 .

APPENDIX I

Standard Solution Concentrations, µg/L

Compound	STD1	STD2	STD3	STD4	STD5
alpha-BHC	5.0	10.0	20.0	40.0	80.0
beta-BHC	5.0	10.0	20.0	40.0	80.0
delta-BHC	5.0	10.0	20.0	40.0	80.0
gamma-BHC (Lindane)	5.0	10.0	20.0	40.0	80.0
alpha-Chlordane	5.0	10.0	20.0	40.0	80.0
gamma-Chlordane	5.0	10.0	20.0	40.0	80.0
Heptachlor	5.0	10.0	20.0	40.0	80.0
Aldrin	5.0	10.0	20.0	40.0	80.0
Heptachlor epoxide	5.0	10.0	20.0	40.0	80.0
Endosulfan I	5.0	10.0	20.0	40.0	80.0
Dieldrin	10.0	20.0	40.0	80.0	160
4,4'-DDE	10.0	20.0	40.0	80.0	160
Endrin	10.0	20.0	40.0	80.0	160
Endosulfan II	10.0	20.0	40.0	80.0	160
4,4'-DDD	10.0	20.0	40.0	80.0	160
Endosulfan sulfate	10.0	20.0	40.0	80.0	160
4,4'-DDT	10.0	20.0	40.0	80.0	160
Methoxychlor	50.0	100	200	400	800
Endrin aldehyde	10.0	20.0	40.0	80.0	160
Endrin ketone	10.0	20.0	40.0	80.0	160
Chlordane - technical					1000
Toxaphene			500		
Aroclor-1016	100	250	500	1000	2000
Aroclor-1221			500		
Aroclor-1232			500		
Aroclor-1242			500		
Aroclor-1248			500		
Aroclor-1254			500		
Aroclor-1260	100	250	500	1000	2000
2,4,5,6-tetrachloro-m-xylene	5.0	10.0	20.0	40.0	80.0
Decachlorobiphenyl	10.0	20.0	40.0	80.0	160

Appendix I (Continued)

Calibration Stock Solutions, mg/L

Compound	Mix	Conc
alpha-BHC	A	5.0
beta-BHC	B	5.0
delta-BHC	B	5.0
gamma-BHC (Lindane)	A	5.0
alpha-Chlordane	B	5.0
gamma-Chlordane	B	5.0
Heptachlor	A	5.0
Aldrin	B	5.0
Heptachlor epoxide	B	5.0
Endosulfan I	A	5.0
Dieldrin	A	10.0
4,4'-DDE	B	10.0
Endrin	A	10.0
Endosulfan II	B	10.0
4,4'-DDD	A	10.0
Endosulfan sulfate	B	10.0
4,4'-DDT	A	10.0
Methoxychlor	A	50.0
Endrin aldehyde	B	10.0
Endrin ketone	B	10.0
Chlordane – technical	TChlor	1000
Toxaphene	Tox	500
Aroclor-1016	AR1660	100
Aroclor-1221	AR1221	100
Aroclor-1232	AR1232	100
Aroclor-1242	AR1242	100
Aroclor-1248	AR1248	100
Aroclor-1254	AR1254	100
Aroclor-1260	AR1660	100

*-The single component pesticides are contained in 2 separate mixes (A and B) when utilizing megabore (0.45mm ID) columns. These same components are combined into 1 mix when utilizing capillary (0.32mm ID) columns.

Breakdown Check Solution (CLP PEM solution), µg/L

Compound	**Concentration**
4,4'-DDT	100
Endrin	50

Surrogate Stock Solution, mg/L

Compound	**Concentration**
2,4,5,6-tetrachloro-m-xylene	10
Decachlorobiphenyl	10

APPENDIX II

Analysis Sequence

Injection	Sample
	hexane rinse
1	breakdown check standard (PEM)
2	ICV standard INDAM
3	ICV standard INDBM
4	ICV standard multi-components(for PCB only analysis - can replace above 3 stds)
5	up to 10 subsequent sample or QC extracts
15	solvent rinse or IBLK (optional - not required)
16	CCV standard INDAM
17	CCV standard INDBM
18	CCV standard multi-components (for PCB only analysis - can replace above 2 stds)
19	up to 10 subsequent sample or QC extracts
last	CCV standard INDAM
last	CCV standard INDBM
last	CCV standard multi-components (for PCB only analysis - can replace above 2 stds)

APPENDIX III

Data Packet Order

I. QC SUMMARY

_____ Analysts 'Client' Comment (hard copy and floppy)
_____ Surrogate Recovery Summary Report
_____ Blank Spike Report
_____ MS/MSD Report
_____ Method Blank Summary

II. SAMPLE DATA:

_____ Organic Analysis Data Sheet
_____ Sample Confirmation Worksheet
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

III. STANDARD DATA:

_____ Linearity Report

Linearity Standards:
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

Continuing Calibration Standards:
_____ CCV Report
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

_____ Method Blank
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Blank Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike Duplicate
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

V. Bench Sheets

_____ Injection Sequence
_____ Target Method
_____ Extraction Bench Sheets
_____ Miscellaneous Work Sheets. i.e. %TS, SDG summary, calculations, HTVR
_____ Standards Logs

VI. Reject Data:

DO NOT COPY DO NOT PAGINATE

Data not used to support sample results.
All data acquired but rejected due to out of control QC.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8081B QA Requirements and Corrective Actions:

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	5 calibration stds for linear, 6 stds for quadratic, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes.	5 calibration stds for linear, 6 stds for quadratic, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes. A second source standard is analyzed to verify the initial calibration.	After performing major instrument maintenance or when persistent difficulties meeting CCV criteria occur.	Recalibrate or narrate faults if possible	Narrative
Initial Calibration Verification	Must be ±15% D for each analyte or averaged across all analytes.	Must be ±15% D for each analyte or averaged across all analytes.	At start of each new analytical sequence, or daily if RTs shift	Rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	Narrative
Continuing Calibration Verification	Must be ±15% D for each analyte or averaged across all analytes.	Must be ±15% D for each analyte or averaged across all analytes.	Every 10 samples or every 12 hours.	Rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	Narrative
Breakdown	Endrin and DDT breakdown must be <=15%	Endrin and DDT breakdown must be <=15%	At the beginning of each sequence.	Rerun, instrument maintenance	Narrative

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	Presence of any target analytes must be below MDL	Must be below ½ the reporting limit	Every analytical batch or 1 per 20 samples	Report quantitated contaminants with a "B" flag, or re-extract if necessary	Case narrative and corrective action form if re-extracted
Surrogate Recovery	Limits to be determined by the lab	Listed in QC database	Every sample	Where contractually required, all surrogates must be within control limits.	Case narrative and corrective action form if re-extracted
Matrix Spike Recovery	Limits not specified	Listed in QC database	Every analytical batch or 1 per 20 samples	If other QC is in control, narrate; otherwise re-extract	Case narrative and corrective action form if re-extracted
MS/MSD RPD	Limits not specified	Listed in QC database	Every analytical batch or 1 per 20 samples	If other QC is in control, narrate; otherwise re-extract	Case narrative and corrective action form if re-extracted
Blank Spike Recovery	Limits not specified	Listed in QC database	Every analytical batch or 1 per 20 samples	If other QC is in control, narrate; otherwise re-extract	Case narrative and corrective action form if re-extracted
Standard Reference Material (SRM) Recovery	None required	None required	Only if requested	Reanalyze or re-extract and reanalyze SRM	Case narrative and corrective action form if re-extracted

APPENDIX V

Compound Elution order on DB5 and DB608 Megabore (0.45 mm ID) Columns

	DB608
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
beta-BHC	gamma-BHC (Lindane)
gamma-BHC (Lindane)	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Isodrin	Isodrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
Endosulfan I	alpha-Chlordane
alpha-Chlordane	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
Endosulfan II	4,4'-DDD
4,4'-DDD	Endosulfan II
Endrin aldehyde	4,4'-DDT
Endosulfan sulfate	Endrin aldehyde
4,4'-DDT	Endosulfan sulfate
Endrin ketone	Methoxychlor
Methoxychlor	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

APPENDIX VI

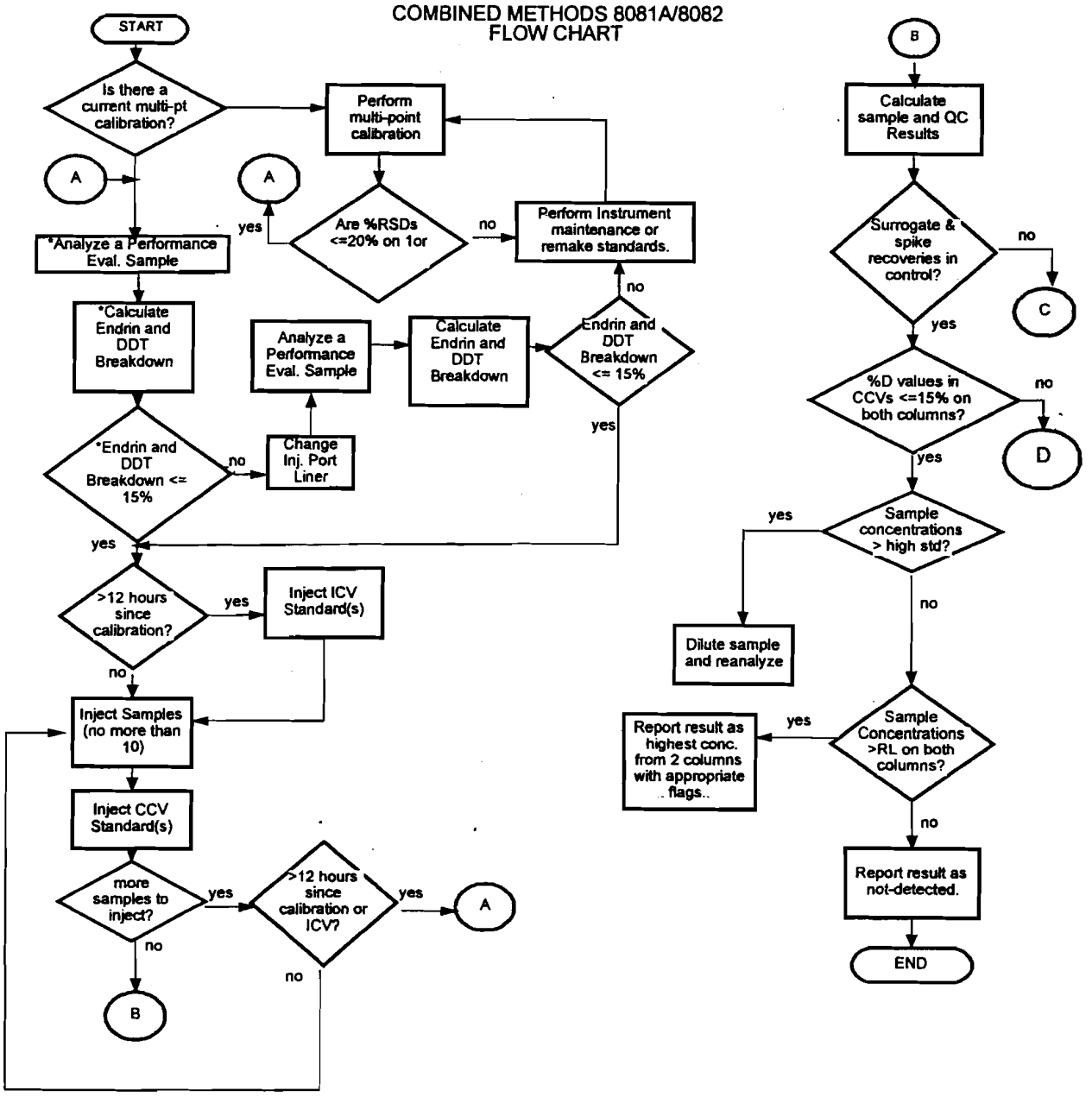
Compound Elution order on DBXLB and DB35ms Capillary (0.32 mm ID) Columns

DBXLB	DB35ms
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
gamma-BHC (Lindane)	gamma-BHC (Lindane)
beta-BHC	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
alpha-Chlordane	alpha-Chlordane
Endosulfan I	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
4,4'-DDD	4,4'-DDD
Endosulfan II	Endosulfan II
Endrin aldehyde	4,4'-DDT
4,4'-DDT	Endrin aldehyde
Endosulfan sulfate	Endosulfan sulfate
Methoxychlor	Methoxychlor
Endrin ketone	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

APPENDIX VII

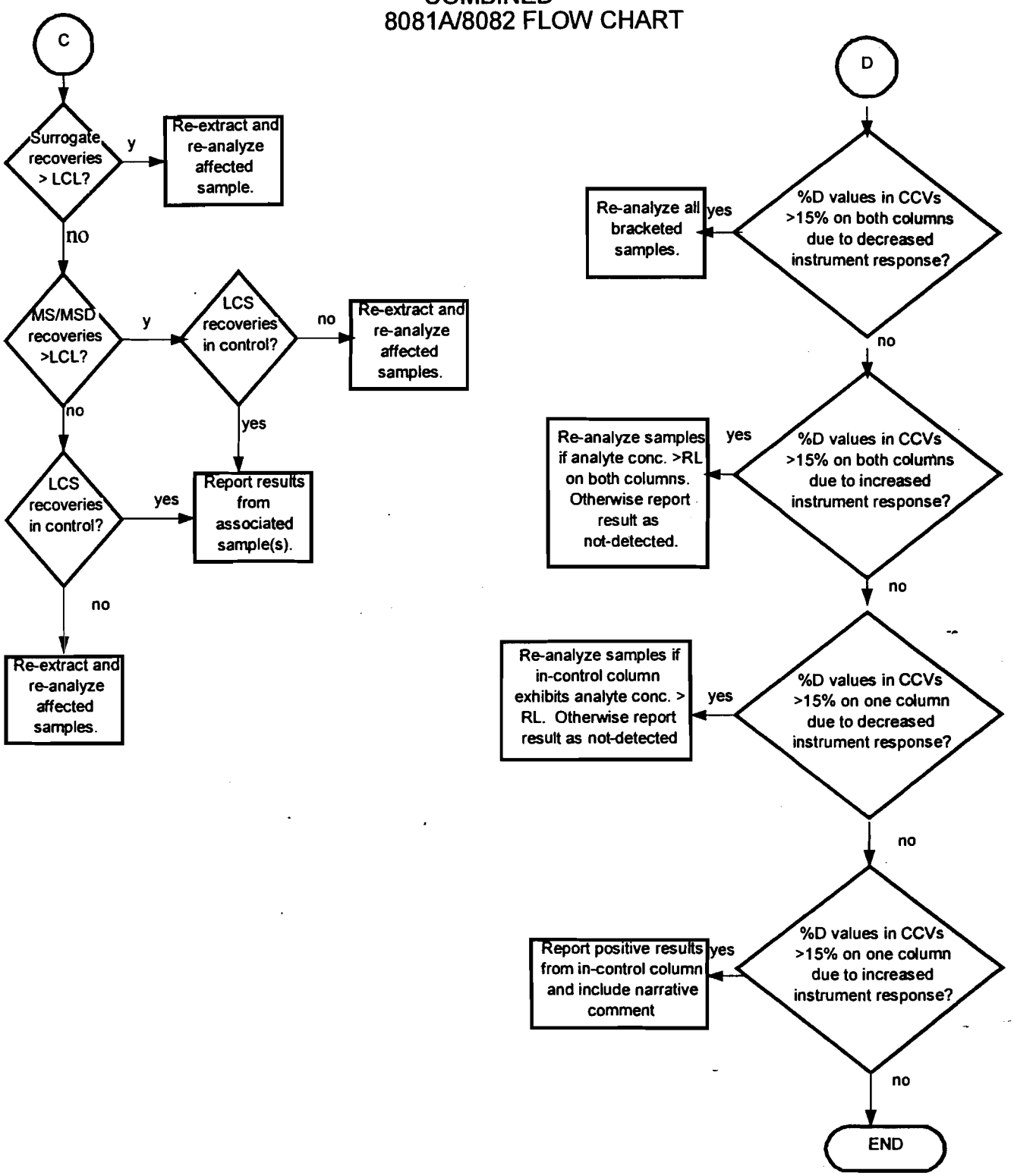
Flow Chart

COMBINED METHODS 8081A/8082
FLOW CHART



*These steps can be eliminated if analyzing for SW8082 compounds only

COMBINED 8081A/8082 FLOW CHART



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8151

Title: **Analysis of Chlorinated Herbicides by Method 8151A**

Revision history:

Number	Date
0	10/08/97
1	2/5/98
2	12/07/98
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Revised by: Monica Carr, Organic Division Manager Date: 3/22/02

Reviewed by: Harry Romberg, QA Officer Date: 3-22-02

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Controlled Document

No. 20 **Assigned to:** TeTr

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method describes the procedures and specifications for instrumental analysis of various chlorinated herbicides in water and soil per Method 8151A in SW-846 utilizing capillary columns. Analysis is performed by gas chromatography using a single injection port and splitting into dual GC columns with electron-capture detectors. This system provides quantitation and confirmation of herbicides from a single injection. Dinoseb generally yields poor recoveries using this analysis due to difficulty in bringing the analyte through the hydrolysis stage of the extraction procedure. The following table lists the compounds that may be determined by this method:

ABBREVIATION	COMPOUND NAME
2,4-D	2,4-Dichlorophenoxyacetic acid
2,4-DB	4-(2,4-Dichlorophenoxy) butyric acid
2,4,5-T	2,4,5-Trichlorophenoxyacetic acid
2,4,5-TP (Silvex)	2-(2,4,5-Trichlorophenoxy) propionic acid
Dalapon	2,2-Dichloropropionic acid
Dicamba	2-Methoxy-3,6-dichlorobenzoic acid
Dichloroprop (2,4-DP)	2-(2,4-Dichlorophenoxy) propionic acid
Dinoseb	2-(sec-Butyl)-4,6-dinitrophenol
MCPA	(4-Chloro-2-methylphenoxy) acetic acid
MCPP	2,4-(Chloro-2-methylphenoxy) propionic acid
NON-ROUTINE COMPOUNDS	
4-Nitrophenol	4-Nitrophenol
PCP	2,3,4,5,6-Pentachlorophenol
Picloram	4-Amino-3,5,6-trichloropicolinic acid
Hexachlorophene	Hexachlorophene

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C. Water samples must be extracted within 7 days of sample collection, soil samples within 14 days of sample collection. All extracts must be analyzed with 40 days of sample extraction.

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier	A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number which is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation.
Blank spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all AIMTech and NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration.
CF	Calibration factor. The ratio of analyte instrument response to nanograms injected. This term is defined in the same way in both the CLP contract and SW 846.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC	Correlation coefficient. A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	Instrument blank. This term is borrowed from CLP. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections.
ICV	Initial calibration verification. This term is borrowed from the CLP GC/MS protocol. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
MDL standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are in the range of 1x to 4x the empirically determined MDLs on an extract/digest basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set.

QC check standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC period	Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RRF	Relative Response Factor. A measure of the relative response of an analyte compared to its internal standard. Relative response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RRF is determined by the following equation: $$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$ Where A = area of response measured C = concentration is = internal standard x = analyte of interest
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.
RT, Retention time	The time (in minutes) at which a target analyte elutes from a chromatography column.
RT window	Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

2.1.1 This analysis requires a gas chromatograph with a programmable oven, heated injection port, dual electron-capture detectors, autosampler and an electronic data-acquisition system.

2.1.2 **Equipment list:**
Gas chromatograph: Hewlett-Packard 5890 or equivalent.
Autosampler: Hewlett-Packard 7673 or equivalent.
Electron-capture detectors: Hewlett-Packard or equivalent.
Capillary chromatographic columns of dissimilar phase: J&W DB5 and DB608 or DB17, 30m x 0.45mm, 0.53mm, or 0.25mm megabore or equivalent.
Helium carrier gas and 5% methane/95% argon detector make-up gas
Data System: EZChrom Data Acquisition System version 6.6, Target Analytical Software for NT Operating System version 4.02, Pentium PCs, or equivalent.

2.2 Standards

2.2.1 Commercially prepared and certified standard solutions are used. Suppliers may change without notice or immediate update to this Standard Operating Procedure. Refer to Laucks' SOP on the traceability, documentation, and preparation standards.

2.2.2 Working standards are prepared in hexane from these stocks. Those which are prepared in the free acid form (not prepared from methyl esters) are methylated by derivatization using diazomethane. At this stage all working standards are in methyl ester or ether form. They must be replaced after six months or by the expiration date of the parent standard, whichever is earlier.

2.2.3 Calibration standards are prepared by combining the stock standards and diluting with hexane to prepare the calibration standard which defines the upper and lower range of the ECD. All calibration standards must be replaced after 6 months or by the expiration date of the parent standard, whichever is earlier.

2.2.4 Appendix I details the compounds and concentrations contained in all solutions.

Primary	Manufacturer	Catalog Number	Comments
8 cmpds	Ultra	HBM-8152M	Methyl Ester
MCPA, MCPP	Restek	32059	Methyl Ester
PCP	Restek	31297	Acid
4-Nitrophenol	Restek	31296	Acid
Picloram	Restek	32266	Methyl Ester
Hexachlorophene	Ultra	PH270-1	Methyl Ester
2,4-DCPAA(surr)	Ultra	PPS-166	Methyl Ester
Second Source	**Manufacturer**	**Catalog Number**	**Comments**
10 cmpds	Supelco	47375	Methyl Ester
PCP	Supelco	48692	Methyl Ester
4-Nitrophenol	Supelco	48695	Methyl Ester
Picloram	Ultra	PS1108	Methyl Ester
Hexachlorophene	Supelco	40323	Methyl Ester
2,4-DCPAA(surr)	Supelco	47339	Methyl Ester

3. Safety precautions and Waste Disposal

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they contain hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 The electron-capture detectors used in this analysis contain a radioactive source and should not be opened or otherwise tampered with. Ensure that all gas cylinders are either fastened to an immovable object or capped securely at all times.

3.2 Waste disposal

3.2.1 Solvent wastes generated in using this procedure (expired standards, old sample extracts, rinsates, etc.) should be emptied into the solvent waste container in the fume hood.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 QA Requirements and Corrective Action

4.1.1 Detailed in the following sections are applicable QA requirements and subsequent corrective actions to be applied to this analysis. A summary of these requirements can be found in Appendix IV.

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on MDL Studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. Current MDLs are listed in the quality control database.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels must be similar to those found in SW-846.

4.3.2 The results must be equal to or better than the spike recoveries tabulated in SW 846. These criteria and recommended spiking concentrations for method validation are tabulated in SW 846.

4.4 Retention Time Windows

4.4.1 Prior to the analysis of any samples, it is necessary to establish retention times windows for the method by analyzing standards for all target analytes over at least a 72-hour period. This need not be a single 72 hour sequence but can be 3 chronologically contiguous sequences. These standards should be inter-mixed with real sample extracts

in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds and compute the standard deviations of all the retention times. Retention time normalization techniques may be applied if appropriate. For megabore capillary columns, a default RT window of ± 0.05 minutes may be used. Details of the procedure for determining retention time windows can be found in Laucks SOP on establishing retention time windows.

4.5 Initial Multi-Point Calibration

4.5.1 Analyze herbicide standards using at least 5 different concentration levels for linear calibrations. The lowest concentration should define the reporting limit (RL). The highest concentration should define the upper usable working range of the detector. Subsequent to the initial calibration, an additional calibration standard is analyzed using standards obtained from a second source. The second source standard must be from a different manufacturer unless one is not available. The acceptance criteria for the second source standard has been defined as ± 20%.

4.5.2 SW 846 allows the use of both linear and non-linear models for the calibration data. The option for non-linear calibration may be necessary to achieve low detection limits or to address specific instrumental techniques.

4.5.3 The choice of a specific calibration model should be made in one of two ways. The first is to begin with the simplest approach, the linear model through the origin, and progressing through the other options until the calibration acceptance criteria are met. The second approach is to use *a priori* knowledge of the detector response to choose the calibration model. Such knowledge may come from previous experience, knowledge of physics of the detector, or specific manufacturer's recommendations.

4.6 Linear Calibration

4.6.1 External standard initial calibration data is evaluated by determining the %RSD of the calibration factors.

4.6.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

4.6.3 The calculated CFs are tabulated and the %RSD calculated. There are no compound-specific criteria. All %RSDs must be within 20% for each analyte or averaged across all analytes in the calibration standard mix. Laucks uses 40% percent as the upper limit for all compounds as an internal guideline although it is not a requirement of SW 846.

4.6.4 Corrective action

4.6.4.1 If the criteria are not met, the instrument must be re-calibrated.

4.7 Non-linear Calibration

4.7.1.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed.

4.7.1.2 When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen should have no more than four parameters, i.e., if the model is polynomial, it may be no more than third order, as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

4.7.1.3 The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches. A quadratic (second order) requires a minimum of six standards, and a third order polynomial requires seven standards. Do not force the line through the origin, i.e., do not set the intercept as 0, and do not include the origin (0,0) as a calibration point. The R^2 value must be greater than 0.900.

4.7.1.4 Whichever option is employed, a surrogate concentration must fall within the calibration range.

4.7.2 Corrective action

4.7.2.1 If the criteria are not met, the instrument must be re-calibrated.

4.8 Initial Calibration Verification

4.8.1 Concentration and/or CF Criteria

4.8.1.1 At the beginning of an analysis sequence analyze a calibration standard. The concentration of the calibration standard is most often the mid-range. However, the concentration of the calibration standard must be varied from time to time. The computed calibration factor (CF) or concentration measurement must meet the criteria detailed below.

4.8.1.2 Using the appropriate calculation technique (average CF) compute either CFs or concentration values. For linear calibrations the ICV standard can be verified by calculating either the percent difference or the percent drift.

4.8.1.2.1 The percent difference calculation compares the ICV CFs to the mean CFs from the initial multi-point calibration. The percent difference is calculated as follows:

$$\%D = \frac{Cv - \overline{CF}}{\overline{CF}} \times 100$$

where:

C_v = Calibration Factor

CF = Mean Calibration Factor

4.8.1.2.2 The percent drift calculation compares the ICV calculated concentrations to the theoretical (or actual) concentration of the ICV standard. The percent drift is calculated as follows:

$$\%D = \frac{C_c - C_T}{C_T} \times 100$$

where:

C_c= Calculated Concentration
C_T= Theoretical Concentration

4.8.1.3 There are no compound-specific criteria. The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.8.2 Corrective action

4.8.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.9 Updating Retention Time Windows

4.9.1 Due to the stability of the ECD, updating retention time windows is not normally required. However, the retention time windows for compound identification can be updated by using the retention times for each target analyte in the ICV standard as the center of the window and the previously determined retention time window half-width to establish the retention time range to be used for compound identification.

4.10 Continuing Calibration Verification

4.10.1 A mid-range calibration standard is analyzed at the frequency of every 10 samples or 12 hours. In addition, this standard must be the last injection made in the analysis sequence.

4.10.2 Criteria

4.10.2.1 After every 10 sample extract injections, or 12 hours, a CCV standard is analyzed. The CF or concentration for each analyte is calculated and the percent difference or percent drift is calculated as shown above.

4.10.3 If the response (or calculated concentration) for an analyte is ±15% of the response obtained during the initial calibration, then the initial calibration is considered still valid. Alternatively, if the average of the responses for all analytes is within 15%, then the

calibration has been verified. However, the average must include all analytes in the calibration, regardless of whether they are target analytes for a specific project, and the data must be provided with the calibration verification data.

4.10.3.1 The retention times for all target analytes must fall within the RT windows established by the ICAL.

4.10.4 Corrective action

4.10.4.1 Check calculations or perform instrument maintenance. To validate the quantification of target analytes in analytical samples, the samples must be bracketed by in-control CCVs. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the RL in the associated samples.

4.11 Method Blanks

4.11.1 Criteria

4.11.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or one blank every 20 samples which ever is more frequent. Any analyte response above 1/2 the reporting limit is reported. Method blank control limits are detailed in the quality control database.

4.11.2 Corrective action

4.11.2.1 Corrective action may necessitate re-extraction of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then the sample group may not require re-extraction. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.12 Method Blank Spikes

4.12.1 Criteria

4.12.1.1 A method blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a method blank spike. A method blank

spike is similar to a QC check standard. Method blank spike recoveries must meet the criteria specified in the quality control database.

4.12.2 Corrective action

4.12.2.1 The method blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re- extraction and re-analysis would be triggered by an out of control method blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

4.13 Matrix Spike

4.13.1 Criteria

4.13.1.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{SS - S}{SA} \times 100$$

where:

SS = spiked sample result
S = unspiked sample result
SA = amount spiked

4.13.1.2 The recovery criteria are detailed in the QC database. In the instance that the native target analyte concentration is greater than 5x the spike added concentration, the MS recovery control limits do not apply. In this case, treat the MS/MSD pair as duplicates and report them as such in the quality control database.

4.13.2 Corrective action

4.13.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.14 Matrix Spike Duplicate

4.14.1 Criteria

4.14.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values.

4.14.1.2 This calculation is as follows:

$$\% RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} \times 100$$

where:

S_1 = measured concentration for MS sample
S_2 = measured concentration for MSD sample

4.14.1.3 RPD control limits are detailed in the QC database.

4.14.2 Corrective action

4.14.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

4.15 Surrogate Recovery

4.15.1 Criteria

4.15.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\% \text{ recovery} = \frac{S_m \times 100}{S_a}$$

where:

S_m = concentration of surrogate measured in extract
S_a = concentration of surrogate added

4.15.1.2 Detailed surrogate recovery control limits are tabulated in the laboratory QC database.

4.15.2 Corrective Action

4.15.2.1 Check calculations for possible error. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recovery since non-GC/MS methods cannot separate co-eluting interferences. Hence corrective action is not usually required for high surrogate recoveries. These cases should be evaluated for other possible causes (such as double surrogate spiking or incorrect dilution factors).

4.15.2.2 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and re-analyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

5. Operation procedures

5.1 Chromatographic Conditions

5.1.1 The following are typical operating parameters used to perform this method. There may be times when adjustments are made in order to optimize the analysis.

Column flow	7-8 mL/min
Make-up gas	argon/methane (95%/5% High Purity Grade)
Make-up flow	70 mL/min
Injector	grob-type, split/splitless with 4mm wide bore deactivated liner
Injector temperature	205°C
Injection	on-column
Injection volume	1µl per column (2µl total)
Initial temperature	45°C
Initial hold time	1.0 min
Temperature ramp	25°C per min to 150°C
Hold time	1.0 min
Temperature ramp	5°C per min to 240°C
Hold time	0 min
Temperature ramp	25°C per min to 290°C
Final hold time	0 min
EC detectors temperature	325°C

5.2 Sample Analysis

5.2.1 Analysis sequence

5.2.1.1 See Appendix II for a detailed analysis injection sequence.

5.2.2 Compound Identification

5.2.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an

observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes. If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

5.2.3 Compound Quantitation

Target compound concentrations are calculated using the following equations:

5.2.3.1 Aqueous samples

The external standard equation, as expressed in SW 846 is:

$$\text{Concentration } (\mu g / L) = \frac{As \times Vt \times D}{\overline{CF} \times Vi \times Vs}$$

where:

A_S = Area or height of the peak for the analyte in the sample.
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was diluted 10 µL to 1000 µL and subsequently diluted an additional 10 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000.
If no dilution was made, D = 1.
V_t = Volume of total extract, µL.
V_S = Initial sample size, mL.
$\overline{CF}$ = Mean calibration factor.

To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/L * 0.001

5.2.3.2 Non-aqueous samples

The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Conc.(\mu g / kg) = \frac{Vt \times As \times D}{CF \times Vi \times Ws}$$

where:

Ws = Weight of sample extracted or purged, grams. Either the wet weight or dry weight..

6. Reports

6.1 Data Packet Organization

See Appendix III for a check list detailing data packet organization.

6.2 Quality Control Reports

All results for quality control tests are entered into the lab quality control data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the reporting limit indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
P	When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. Generally, we report the lower value.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
C	The target analyte's presence was confirmed by GC/MS.

7. References

1) *SW846 Test Methods for Evaluating Solid Waste*, Method 8000B Gas Chromatography, US EPA Office of Solid Waste and Emergency Response, Revision 2, December 1996.

2) *SW846 Test Methods for Evaluating Solid Waste*, Method 8151A Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzylation Derivatization, US EPA Office of Solid Waste and Emergency Response, Revision 1, December 1996.

APPENDIX I

Calibration Levels, µg/mL

Compound	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6	Level 7	RL Water (µg/L)	RL Soil (ug/kg)
2,4-D	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
2,4-DB	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
2,4,5-T	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
2,4,5-TP (Silvex)	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
Dicamba	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
Dichlorprop (2,4-DP)	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
Dinoseb	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
Dalapon	0.008	0.016	0.040	0.080	0.160	0.200	0.400	.08	2.67
2,4-DCPAA(surr)	0.008	0.016	0.040	0.080	0.160	0.200	0.400	N/A	N/A
MCPA	4.00	8.00	20.00	40.00	80.00	100.00	200.00	40	1334
MCPP	4.00	8.00	20.00	40.00	80.00	100.00	200.00	40	1334
Hexachlorophene	0.05	0.10	0.25	0.50	1.00	5.00	10.00	0.5	16.7

At the time of this writing these are the current calibration concentration levels. However, these levels may change due to project-specific requirements, or a change in standard suppliers.

Spiking Compounds

2,4-D	Dichlorprop (2,4-DP)
2,4-DB	Dinoseb
2,4,5-T	Dalapon
2,4,5-TP (silvex)	MCPA
Dicamba	MCPP

APPENDIX II

Analysis Sequence

Injection	Sample
	rinse
1	ICV standard
2	up to 10 subsequent sample or QC extracts
12	CCV standard
13	up to 10 subsequent sample or QC extracts
last	CCV standard

APPENDIX III

Data Packet Sequence

I. QC SUMMARY

Surrogate Recovery Summary Report
Blank Spike Report
MS/MSD Report
Method Blank Summary

II. SAMPLE DATA:

Organic Analysis Data Sheet
Sample Confirmation Worksheet
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column

III. STANDARD DATA:

Initial Calibration Verification
Linearity Report
Surrogate RT Evaluation Report
CCV Reports
Confirmation Reports

Linearity Standards:
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column

Continuing Calibration Standards:
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column
Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

Method Blank
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column

Blank Spike
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column

Matrix Spike
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column

Matrix Spike Duplicate
Chromatograms, primary column
Chromatograms, secondary column
Chromatographic Report, primary column
Chromatographic Report, secondary column

V. Bench Sheets

Injection Sequence
Extraction Bench Sheets
Miscellaneous Work Sheets. i.e. %TS, SDG summary, calculations, HTVR
Standards Logs

VI. Reject Data:

Data not used to support sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8151 QA Requirements and Corrective Actions:

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	5 calibration std. for linear, 6 std. for quadratic, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes.	5 calibration std. for linear, 6 std. for quadratic, for all single and multi-components, %RSD ≤20% for each analyte or averaged across all analytes. Verification using a 2nd source ± 20%.	After performing major instrument maintenance or when persistent difficulties meeting CCV criteria occur.	recalibrate or narrate faults if possible	Narrative
Initial Calibration Verification	Must be ±15% D for all quantitated analytes (or average of %Ds ±15%).	Must be ±15% D for all quantitated analytes (or average of %Ds ±15%).	once per daily analytical sequence	rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	Narrative
Continuing Calibration Verification	Must be ±15% D for all quantitated analytes (or average of %Ds ±15%).	Must be ±15% D for all quantitated analytes (or average of %Ds ±15%).	Every 10 samples or every 12 hours.	rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible)	Narrative
Method Blank	Must be below MDL	Must be below 1/2 reporting limit	every analytical batch or 1 per 20 samples	Report quantitated contaminants with a "B" flag, or re-extract if necessary	case narrative and corrective action form if reextracted

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Surrogate Recovery	Limits to be determined by lab	listed in QC database	every sample	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
Matrix Spike Recovery	every analytical batch or 1 per 20 samples, limits to be determined by lab	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
MS/MSD RPD	every analytical batch or 1 per 20 samples, limits to be determined by lab	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted
Blank Spike Recovery	every analytical batch or 1 per 20 samples, limits to be determined by lab	listed in QC database	every analytical batch or 1 per 20 samples	if other QC is in control, narrate; otherwise re-extract	case narrative and corrective action form if re-extracted

APPENDIX V

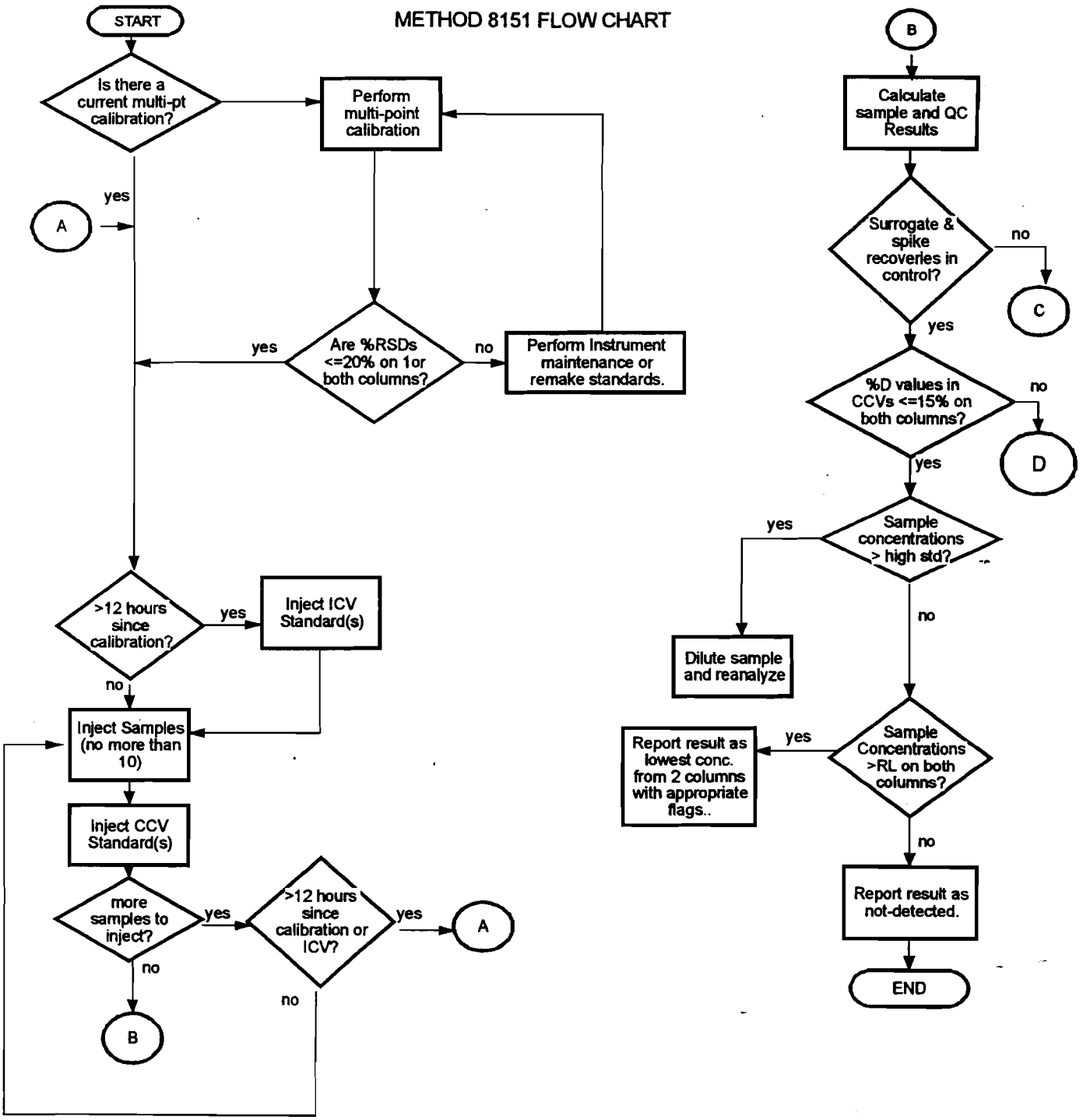
Compound Elution Order

Column DB-5	Column DB-608
Dalapon	Dalapon
4-Nitrophenol	4-Nitrophenol
2,4-DCPAA (surr)	2,4-DCPAA (surr)
Dicamba	MCPP
MCPP	Dicamba
MCPA	MCPA
Dichloroprop (2,4-DP)	Dichloroprop (2,4-DP)
2,4-D	2,4-D
Pentachlorophenol	Pentachlorophenol
2,4,5-TP (Silvex)	2,4,5-TP (Silvex)
2,4,5-T	2,4,5-T
2,4-DB	Dinoseb
Dinoseb	2,4-DB
Picloram	Picloram
Hexachlorphene	Hexachlorphene

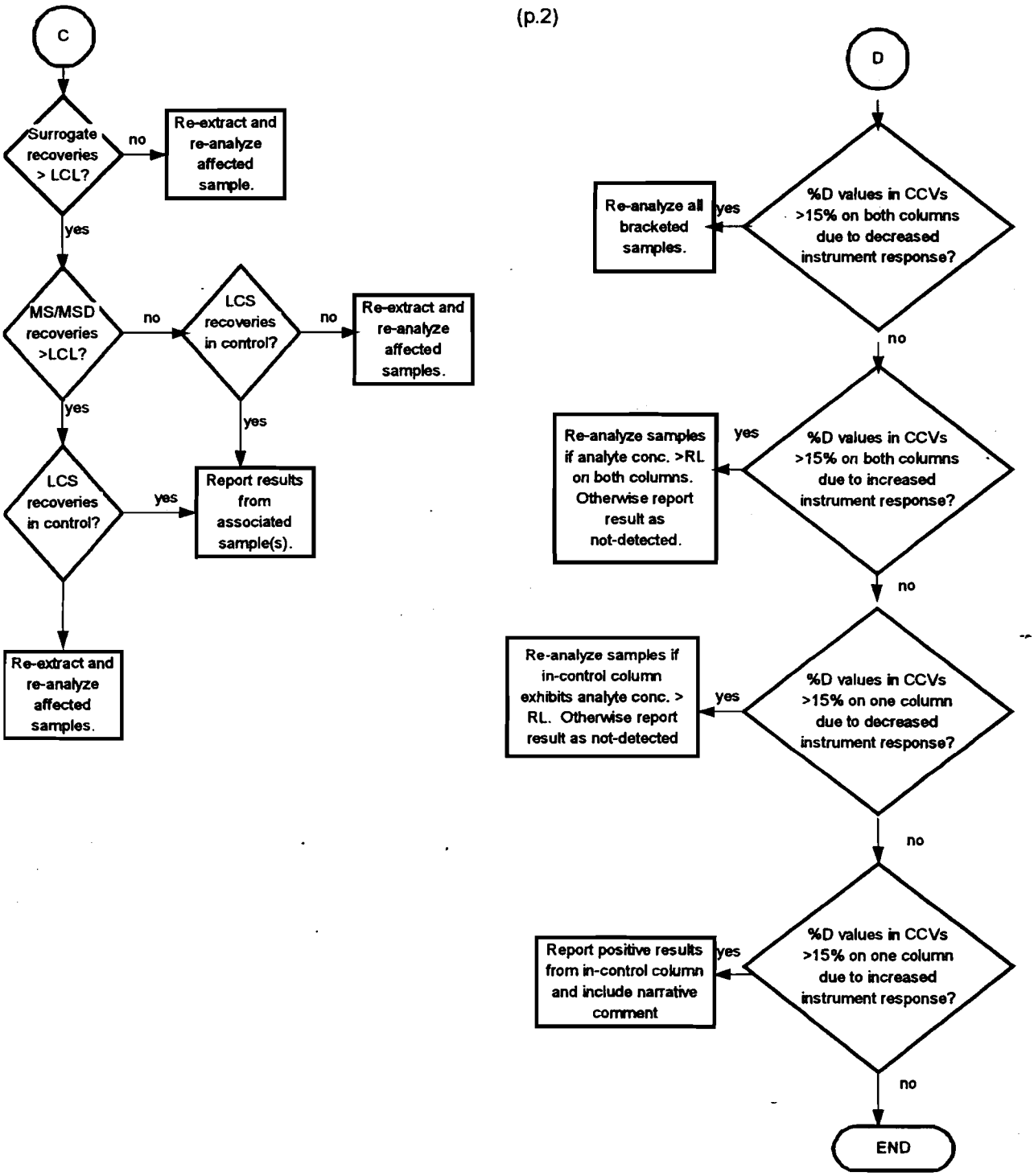
APPENDIX VI

Flow Chart

METHOD 8151 FLOW CHART



METHOD 8151 FLOW CHART
(p.2)



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8265

Title: **Determination of Volatile Organic Compounds by Method SW 8260B**

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3) The BFB tuning criteria used is taken from CLP SOW OLM04.2, which is not a deviation from the method since SW 8260B allows the use of alternative tuning criteria, e.g., CLP and/or 524.2 The tuning criteria are tabulated in section 4.4.5.

1.3 Interferences

1.3.1 Problems have been associated with the following compounds analyzed by this method.

1.3.1.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

1.3.1.2 Bromoform is one of the compounds to most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

1.3.1.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in the purge-and-trap system and/or active sites in trapping materials.

1.4 Sample Collection, Sample Storage, Holding Times

1.4.1 Waters

1.4.1.1 Samples are collected with zero headspace in 40 mL glass containers containing preservative (either HCl, H_2SO_4 or solid $NaHSO_4$) and sealed with Teflon-lined caps.

1.4.1.2 The water samples are preserved to a pH of ≤2, and all samples are stored at 4°C.

1.4.1.3 The holding time to analysis is 14 days from date of collection. The pH values for the water samples are measured subsequent to sample analysis. The pH values, when not preserved to a pH ≤2 are noted in the instrument logbook and the client report.

1.4.2 Soils

1.4.2.1 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that low level soils be collected in duplicate using 5 gram and/or 1 gram aliquots which are to be weighed out in the field. The soil samples are to be collected in a 40 mL vial containing water, sodium bisulfate, and a stir bar. 0.2 grams of sodium bisulfate are added for each 1 gram of soil (see section 1.4.2.2 for issues associated with this method). Therefore, when collecting 5 grams of sample, the VOA vial should contain 1 gram of sodium bisulfate. The sodium bisulfate is added in order to ensure a sample pH of ≤2. The vials

used for low level soils should have the tared weight recorded on the label and sealed with septum-lined screw caps.

1.4.2.2 An in-depth study of the different sampling methods used for Method SW 846 5035 was performed by the IT Corporation and presented to the EPA at it's annual conference in May 2001.

1.4.2.3 Problems associated with sodium bisulfate were confirmed, including styrene degradation, acetone formation, and effervescence in some samples. Dangerous pressure and high methyl acetate results were also noted with sodium bisulfate.

1.4.2.4 IT is recommending removal of the sodium bisulfate preservation option from the next CLP SOW and EPA is planning to follow this recommendation

1.4.2.5 Another option is the use of the EnCore™ sampler, the Purge-and-Trap Sampler™, and a cut plastic syringe to transfer the sample to the soil vial. If the EnCore™ sampler is used in the field, the samples may be stored for up to 48 hours. However, samples collected in this device should be transferred to the soil sample vials (and preserved) as soon as possible, or analyzed within 48 hours. The holding time for soils is 14 days from the date of collection.

1.4.2.6 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that high level soils be collected in using one of the following options:

1.4.2.6.1 Option 1: Samples will be collected in duplicate using 5 gram and/or 1 gram aliquots which are to be weighed out in the field. The soil samples are to be collected in a 40 mL vial containing methanol. The vials used for high level soils should have the tared weight recorded on the label and sealed with septum-lined screw caps.

1.4.2.6.2 Option 2: Samples will be collected using an EnCore type sampler or equivalent, and preserved at the laboratory by freezing.

1.4.2.6.3 Option 3: Samples will be collected in vials with zero headspace.

1.4.2.7 Per client request Laucks will continue to accept unpreserved soil samples for low or high level analysis. The samples must be collected in 2- or 4-oz glass containers and sealed with teflon-lined caps. These samples will be introduced by method 5030A and analyzed according to this SOP. Please note that for South Carolina samples, Method 5035 must be followed.

1.4.2.8 The holding time to analysis is 14 days from date of collection.

1.5 Definition of Terms

1.5.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time samples are analyzed. Blank spikes are required on all NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCC	Calibration check compound. There are 6 analytes which must not exceed the maximum %RSD of 30% in the initial calibration and a %D of 20% in the continuing calibration standard to validate linearity. These analytes are 1,1-dichloroethane, chloroform, 1,2-dichloropropane, toluene, ethylbenzene and vinyl chloride.
CCV	Continuing calibration verification. This is a standard analyzed at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
IBLK	Instrument blank. An instrument blank is PFW containing the method surrogates and is introduced into the instrument to monitor for carry over between sample analyses.
ICV	Initial continuing verification. A calibration standard obtained from a different manufacturer than the standards used for the initial and continuing calibrations that is analyzed after a compliant initial calibration.

IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
PFW	Purge-free water. Laboratory deionized water that is boiled for 30 minutes prior to use. The systems used to provide deionized water at Laucks all contain carbon polishing filters which are capable of providing purge-free water for use as reagent water.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of BFB, followed by the standard. A QC period is terminated after 12 hours from the injection of BFB.
Reporting Limit (RL)	The reporting limit is defined by the low level standard, also referred to as the practical quantiation limit (PQL).
RF	Response factor. A measure of the response of an analyte compared to its internal standard response. Response factors are determined by standard analysis and are used in the calculation of concentrations of analytes in samples.
RRT	Relative retention time. A measure of the retention time of an analyte compared to the retention time of its internal standard.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

RT	Retention time. The time (in minutes) at which a target analyte elutes from the GC column.
Sequence	A set of samples and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.
SPCC	System performance check compound. Specified compounds in which the minimum RFs must be met in order to demonstrate that the initial and continuing calibration standards are in control. These compounds are chloromethane, 1,1-dichloroethane, bromoform, chlorobenzene, and 1,1,2,2-tetrachloroethane.

2. Equipment List and Standards

2.1 Chromatographic System

2.1.1 Gas Chromatograph: Hewlett Packard 5890 I or II or 6890 employing a low-dead-volume interface from the sample concentrator to the GC injection port.

2.1.2 Carrier Gas: Helium 99.995% (high purity grade) or better.

2.1.3 Column: 60m x 0.25mm x 1.4 µm film thickness, or 30 m x 0.53 mm x 3.0 µm film thickness or 75 m x 0.45 x 2.55 µm film thickness, fused silica capillary column (J&W DB-624 or Restek RTx-624 or equivalent).

2.1.4 Purge and Trap: OI Analytical 4460A and 4560 sample concentrators or equivalent.

2.1.5 Autosampler: Dynatech Precision Sampling PTA-30W/S, Varian Archon or equivalent.

2.1.6 GC/MS Interface: Hewlett Packard 59913A jet separator assembly, split/splitless or equivalent.

2.1.7 Mass Spectrometer: Hewlett Packard 5970B, 5973 or equivalent.

2.1.8 Data System: PC based Teknivent with ENVIROLINK data acquisition and processing software or HP Chemstation.

2.1.9 Trap: OI Corporation, Supelco, or equivalent. Examples are listed below:

1) 1 cm 3% SP-2100/Chromosorb W AW, 7.7 cm Tenax TA, 7.7 cm silica gel 15, 7.7 cm activated charcoal. Supelco Pt No.:2-1139.

2) VOCARB 3000. 10 cm Carbopack C, 6 cm Carboxen 1000, 1 cm Carboxen 1001. Supelco Pt No.: 2-1131.

3) VOCARB 4000. 8.5 cm Carbopack C, 10 cm Carbopack B, 6 cm Carboxen 1000, 1 cm Carboxen 1001. Supelco Pt. No.: 2-1143.

2.1.10 Miscellaneous: Assorted gas-tight calibrated syringes, pipettes, 40 mL vials, caps, septa, sea sand, purge and trap grade methanol, ferrules, pH strips, purge-free DIW, volumetrics, laboratory oven capable of heating glassware up to 100°C, and assorted supplies.

2.2 Standards

2.2.1 Overview of Volatile Standards Preparation

2.2.1.1 All standards are made using a high purity purge and trap grade methanol. Every lot number of methanol is analyzed prior to use to ensure that it is free of volatile organic contaminants. A record of analysis of an aliquot from each batch is stored for a minimum of one year.

2.2.1.2 Two standard logbooks are employed to document all volatile standards. One standard logbook is maintained for stock solutions prepared from neat materials and to record the login of certified standards. The other standard logbook is maintained for working solutions which include solutions procured from commercial sources, as well as those solutions made in the laboratory. All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation of standards.

2.2.1.3 All standards must be identified and quantitated prior to use in the laboratory. This is performed by purchasing certified standard solutions from a commercial supplier that is recognized by the EPA and which are traceable to NIST. In addition, standards for the permanent gases and non-gases are monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if the calibration standard(s) fail to meet the criteria specified in SW 846. Standards for gases need to be replaced after one week, or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the

standard can be documented. If the standards meet the criteria for the calibration standard(s) as specified in SW 846, they are considered acceptable.

2.2.1.4 All standard solutions are stored in the VOA freezer at -10°C to -20°C. The stock and secondary dilution standards are stored in flame-sealed amber ampules (with the exception of the internal and surrogate standards). The secondary dilution standards are checked for degradation or evaporation prior to preparing calibration standards. Secondary dilution standards should be replaced after one week, unless the acceptability of the standard can be documented. Pre-mixed certified solutions are stored according to the manufacturers' documented holding time and storage temperature recommendations. Prior to daily and initial calibration standard preparation, the standard solutions are removed from the freezer and allowed to warm to room temperature.

2.2.1.5 Refer to Appendix I for a tabular listing of all standard solutions and their concentrations. The additional appendix IX compounds (which are only analyzed upon request) and their standard concentrations are tabulated in Appendix IV.

2.2.2 Preparation of Internal Standards and Surrogate Standards

2.2.2.1 If starting with a neat material, a measured amount of the neat chemical is placed in a glass volumetric and diluted to volume with methanol, then stoppered. 125 mg of neat material is diluted into 25 mL of methanol. This yields an intermediate solution of 5000 µg/mL.

2.2.2.2 Aliquots of the intermediate solutions are taken from the individual internal and surrogate standards and then combined in a volumetric flask and diluted with methanol to yield a working standard which contains all compounds at a concentration of 250 µg/mL each. This is accomplished by using a gas-tight syringe to transfer each aliquot of 1.25 mL into 25 mL of methanol. A 1 µL aliquot of this solution is injected via the autosampler into 5 mL for waters, low and medium level soils (and 25 mL for low level waters) of sample, resulting in an internal and surrogate standard concentration of 50 µg/L (or 10 µg/L for low level waters).

Internal Standards	Surrogate Standards
Fluorobenzene	1,2-Dichloroethane-d4
Chlorobenzene-d5	Toluene-d8
1,4-Dichlorobenzene-d4	p-Bromofluorobenzene
	Dibromofluoromethane

2.2.3 Preparation of Matrix Spike Solution

1. Introduction and Scope

1.1 Method Description

Method 8260 is used in the determination of volatile organic compounds in soil, sediment, aqueous, and other matrices. This SOP addresses the determination of volatile organics in different matrices, which include medium level soils as well as low level water samples. A 25 mL sample volume is employed in order to achieve lower detection limits for the low level waters. In addition, the instrument acquisition parameters have been added for the analysis of vinyl chloride by Selected Ion Monitoring (SIM) in Appendix III, and Appendix IV contains a tabular list of the non-routine Appendix IX analytes and their respective calibration ranges.

1.1.1 Because this method employs sample introduction via a purge and trap sample concentrator, it is applicable to volatile compounds that have boiling points below 200°C and that are insoluble or only slightly soluble in water. Compounds that are more soluble in water have elevated detection limits due to their decreased purging efficiency. These include, but are not limited to ketones, nitriles, acetates, acrylates and ethers.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the use of purge and trap systems, gas chromatography/mass spectroscopy and in the interpretation of chromatograms and mass spectral data. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and data interpretation.

1.2 Method Deviations

1.2.1 Detailed below are any deviations from the published version of method SW 8260B. All deviations are followed as the standard operation procedure by Laucks Testing Labs.

1) Laucks uses 1/2 the reporting limit (low level standard) for method blank criteria instead of method detection limits as referenced in SW 846. Common laboratory solvents, such as methylene chloride and acetone, are allowed to be present in the method blanks up to the RL as well as bromomethane in medium soil analyses. Refer to Section 6.4 for detailed method blank criteria.

2) The retention time window used for internal standards in all analyses is +/- 0.50 minutes relative to the daily CCV standard.

2.2.3.1 At a minimum, five volatile analytes are used for spiking. Upon request by the client additional analytes may be used if they are of interest at the site. A certified matrix spiking solution which contains the five spiking analytes is purchased pre-mixed from a commercial supplier at a concentration of 2500 µg/mL each. This solution is then diluted in methanol to a concentration of 200 µg/mL.

Matrix Spike Solution	Source*	Stock Number*
1,1-Dichloroethene	Restek	30005
Benzene		
Trichloroethene		
Toluene		
Chlorobenzene		

* or equivalent

2.2.3.2 A 12.5 µL (2.5 µL for low level waters) aliquot of this solution is spiked into 50 mL of sample, resulting in a spiked concentration of 50 µg/L (or 10 µg/L for low level waters) for each analyte.

Working Concentration	Aliquot	Final Volume	Concentration
Soils, 5 mL Water			
200 µg/mL	12.5 µL	50 mL PFW	50 µg/L
Medium Level Soils			
200 µg/mL	12.5 µL	50 mL PFW	(50 µg/L extract) 5000 µg/L sample
25 mL Water			
200 µg/mL	2.5 µL	50 mL PFW	10 µg/L

2.2.4 Preparation of p-Bromofluorobenzene (BFB) Solution

2.2.4.1 The preparation of BFB is similar to that of the internal standards and surrogate standards. Refer to Section 2.2.2 for details.

2.2.4.2 A solution of 25 ng/mL is prepared and stored in flame-sealed amber ampules.

2.2.5 Preparation of Initial Calibration Standards

2.2.5.1 Certified sets of stock solutions which contain all target analytes are purchased pre-mixed from a commercial supplier at a concentration of 2000 µg/mL each. If additional target analytes are requested, these analytes may be ordered separately, either in solution or as a neat material.

2.2.5.2 These solutions are then diluted in methanol to generate standards at concentrations of 200 µg/mL. This solution is stored in flame-sealed amber ampules.

2.2.5.3 Standards are stored in the VOA freezer at -10°C to -20°C. They are removed prior to making up the initial calibration standards and are allowed to warm to room temperature.

2.2.5.4 The initial calibration standards range from 1 µg/L to 50 µg/L for low level waters and 3 µg/L to 200 µg/L for soils, and 5 mL water samples for all target analytes with the exception of the ketones and m,p-xylenes. Please refer to Appendix I for a tabular listing of all analytes and calibration concentrations. The 5 mL water calibration standards are also employed in the analysis of medium or high level soils, since the aqueous calibration standards are purged at ambient temperature. Analysis of the solid matrices in the low level format requires a purge temperature of 40° C.

2.2.5.5 The standards are prepared by taking aliquots of the solutions and diluting them into a volumetric flask containing PFW. A 40 mL aliquot is immediately poured into a VOA vial (or a 5 mL aliquot for the low level soils is placed in a soil vessel) and placed on the autosampler. Each standard concentration is prepared just prior to analysis and any remaining standard solution is disposed of once the analytical run is initiated.

Solution	Source*	Stock Number*
VOC Mix 1	Supelco	4-8775
VOC Mix 2	Supelco	4-8777
VOC Mix 3	Supelco	4-8779
VOC Mix 4	Supelco	4-8786
VOC Mix 5	Supelco	4-8797
VOC Mix 6	Supelco	4-8799
TCL Mix 1	Supelco	4-8949
Carbon Disulfide	Supelco	4-0363

* or equivalent

Low Level Water (25 mL)

Working Solution	Aliquot non-ketones	Aliquot ketones	Final Volume	Concentrations non-ketones	Concentrations ketones
200 µg/mL	1.0 µL	-	200 mL	1 µg/L	
200 µg/mL	2.5 µL	2.5 µL	100 mL	5 µg/L	5 µg/L
200 µg/mL	5.0 µL	5 µL	100 mL	10 µg/L	10 µg/L
200 µg/mL	12.5 µL	12.5 µL	100 mL	25 µg/L	25 µg/L
200 µg/mL	12.5 µL	12.5 µL	50 mL	50 µg/L	50 µg/L
200 µg/mL	-	25 µL	50 mL	-	100 µg/L

Low Level Soil

Working Solution	Aliquot non-ketones	Aliquot ketones	Final Volume	Concentrations non-ketones	Concentrations ketones
200 µg/mL	1.5 µL	2.5 µL	100 mL/50 mL	3 µg/L	10 µg/L
200 µg/mL	2.5 µL	5 µL	50 mL	10 µg/L	20 µg/L
200 µg/mL	12.5 µL	12.5 µL	50 mL	50 µg/L	50 µg/L
200 µg/mL	5.0 µL	5.0 µL	10 mL	100 µg/L	100 µg/L
200 µg/mL	10.0 µL	10.0 µL	10 mL	200 µg/L	200 µg/L

5 mL Water And Medium Level Soil

Working Solution	Aliquot non-ketones	Aliquot ketones	Final Volume	Concentrations non-ketones	Concentrations ketones
200 µg/mL	1.5 µL	2.5 µL	100 mL/50 mL	3 µg/L	10 µg/L
200 µg/mL	2.5 µL	5 µL	50 mL	10 µg/L	20 µg/L
200 µg/mL	12.5 µL	12.5 µL	50 mL	50 µg/L	50 µg/L
200 µg/mL	25 µL	25 µL	50 mL	100 µg/L	100 µg/L
200 µg/mL	50 µL	50 µL	50 mL	200 µg/L	200 µg/L

2.2.6 Preparation of a Daily Calibration Standard

2.2.6.1 The concentration of the continuing calibration standard at a level of 10 µg/L for low level waters, 50 µg/L for low and medium level soils and 5 mL waters, is also the mid-point of the initial calibration curve. This standard is always prepared just prior to analysis by transferring an aliquot of each of the 200 µg/mL multi-component working solutions into a volumetric containing PFW.

3. Safety precautions & Waste disposal

3.1 Routine Safety Precautions

3.1.1 All standards and samples should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Flammable solvents such as methanol are stored in the appropriate solvent locker located outside the volatile laboratory.

3.1.6 Many analytes are known or suspected carcinogens. Analysts should take the proper precautions such as wearing gloves when working with suspect samples or high level standards or solvents. Additionally, a respirator should be worn and a fume hood utilized for extremely hazardous compounds.

3.2 Waste disposal

3.2.1 Waste solvents and expired standards are disposed of in the appropriate waste solvent container located in the prep area under the hood.

3.2.2 Solid sample matrices are disposed of in appropriate drums labeled for this specific purpose where they are routinely picked up by an approved agency for final disposal.

4. Operation procedures

4.1 Analytical Conditions

GC Parameters	5970	5973
Injection Port Temperature	: 200°C	250°C
GC/MS Interface Temperature	: 250°C	250°C
Initial GC Temperature	: 35°C	40 °C
Initial Hold Time	: 4 min	4 min
Ramp Rate	: 4°C/min	10°C/min
Final Temperature	: 140°C	200°C
Final Time	: 4 min	8 min
Carrier Gas Flow	: 15 mL/min	
Column Head Pressure	: 9 psi	

Purge and Trap Parameters

Purge Time	: 11 min	11 min
Desorb Time	: 1 min	2 min
Bake Time	: 16 min	8 min
Desorb Preheat Temperature	: 20°C	180°C
Desorb Temperature	: 180°C	250 °C
Purge Flow	: 40 mL/min	40 mL/min
Purge Temperature	: Ambient (40°C for soils)	Ambient (40°C for soils)
Desorb Flow Rate	: 15 mL/min	15 mL/min

Mass Spectrometer Parameters

Electron Energy	: 70 volts
Mass Range	: 35 to 300 amu
Scan Time	: 1.4 sec/scan
Scan Start Time	: 0.1 min

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP for the determination of method detection limits. It involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2.2 MDL studies will continue to be performed. This procedure is fully described in LTL-1011, *Procedure for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits.*

4.2.3 MDLs for this method typically are in the range of 0.2-3µg/L for 5 mL waters, 0.3-3.0µg/kg for low level soils and 0.06-1.0 µg/L for 25 mL waters.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically 10-50 times the MDL. In the absence of criteria for the initial demonstration of proficiency, the limits of 70%- 130% can be used.

4.4 Instrument Tuning

4.4.1 Prior to the analysis of any samples, blanks, or calibration standards, the instrument must demonstrate mass calibration and resolution by meeting the established tuning criteria for BFB.

4.4.2 FC-43 (PFTBA) is used as a mass calibrant. The following ratios are suggested in order to meet the tuning criteria for BFB:

Ion	% of ion 69	Isotope	Isotope Abundances
69	100%	70	0.5-1.5%
131	25-40%	132	2.0-4.0%
219	25-40%	220	2.5-5.0%
414	1-3%		

4.4.3 In addition to the tabulated abundances listed above, other criteria should be reviewed at this time.

1) Ion peak widths: The appropriate range is between 0.5 and 0.6 amu. Amu widths beyond this range can lead to loss of minor isotopes, while insufficient peak widths will result in decreased sensitivity.

2) Leak check: The abundances of ions 18, 28, and 32 (water, nitrogen and oxygen, respectively) should be less than 5 percent relative to 69. A level greater than 5 percent is indicative of a leak in the system.

4.4.4 Following the system tuning using FC-43, the GC/MS is then calibrated with BFB. A 2 µL aliquot (1 µL for low level waters) of 25 ng/µL of BFB is injected directly into the GC resulting in a concentration of 50 ng (25 ng for low level waters).

4.4.5 The ion abundances and ratios are checked against the criteria detailed below **or** alternatively, other criteria may be used (e.g., CLP, 524.2). If BFB meets the tuning criteria, then the system is determined to be calibrated. If BFB does not meet the tuning criteria, the GC/MS is re-tuned with FC-43 as described above.

4.4.6 SW 846 allows for the use of 5 - 50 ng of BFB. In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, SW 846 states that the following approach has been shown to be useful: the mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

4.4.7 CLP requires that the three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

SW 846 Tuning Criteria

Mass	Abundance %
50	8 - 40% of mass 95
75	30 - 60% of mass 95
95	base peak, 100% relative abundance
96	5 - 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 - 9% of mass 174
176	>95%, < 101% of mass 174
177	5 - 9% of mass 176

CLP Tuning Criteria

Mass	Abundance %
50	8 - 40% of mass 95
75	30 - 66% of mass 95
95	base peak, 100% relative abundance
96	5 - 9% of mass 95
173	< 2% of mass 174
174	50-120% of mass 95
175	4 - 9% of mass 174
176	93 - 101% of mass 174
177	5 - 9% of mass 176

4.5 Initial Multi-Point Calibration

4.5.1 The first option is to begin with the simplest approach, the linear model through the origin using RSDs, as described in section 7.5 of 8000B. In methods where this method is not appropriate, because the calibration acceptance criteria cannot be met, the next option of calibration (linear regression) is used. This calibration option is typically used in fuels because of the elevated response obtained in these analyses. The requirements for the linear regression model is that R^2 be greater than or equal to 0.99. The more complex calibration option is use of the quadratic curve. This calibration option requires a minimum of 6 points and is used in cases where the detector doesn't meet calibration criteria over a wide range. The difficulty with the quadratic is that the intercept may be

greater than the detection limit. This calibration option exhibits a greater accuracy in the mid-range, and may demonstrate more extreme responses at the low and high end of the calibration, and therefore, it is important to dilute samples and extracts so that they are in the mid-range of the calibration.

4.5.2 For linear calibrations using the RSD, analyze standard solutions using a minimum of 5 different concentration levels. The lowest concentration should establish the reporting limit. The highest concentration should define the upper usable working range of the detector. Criteria for evaluating these standards are detailed in Section 6.

4.5.3 After completion of a compliant initial calibration curve, an initial calibration verification (ICV) may be analyzed. This second source independent calibration verification may be performed by analyzing a LCS as long as it meets the same acceptance criteria as a CCV detailed in Section 6.

4.6 Continuing Calibration Verification

4.6.1 A CCV is analyzed once the BFB tuning criteria have been met and the instrument has been calibrated. Criteria for evaluating a CCV standard are detailed in Section 6.

4.7 Instrument Blank

4.7.1 The analysis of an instrument blank (IBLK) is performed prior to the injection of BFB. This analysis ensures that there are no volatile organic contaminants in either the purge and trap sample concentrator or in the autosampler.

4.7.2 Any sample that demonstrates concentrations of target analytes high enough to saturate the detector must be followed by at least one IBLK, also known as a saturation blank. This IBLK analysis is performed to prevent any possible carry-over into the subsequent sample analysis. Evaluation criteria follow the same guidelines set forth for the analysis of a method blank.

4.8 Method Blank

4.8.1 Immediately following the analysis of the CCV and prior to sample analysis, a method blank must be analyzed. The analysis of the method blank demonstrates that the instrument is free of volatile organic contaminants and ensures that the reagent water is also free of contaminants. Refer to Section 6 for method blank acceptance criteria.

4.9 Sample Analysis

4.9.1 Samples (and associated QC) may be analyzed for up to 12 hours from the injection of BFB. The samples are analyzed subsequent to the calibration standard and method blank analyses. However, no more than twenty sample analyses are performed in a 12 hour period. Once all criteria for BFB, the calibration standard, and method blank have been met, the sample analyses are performed. The aqueous and medium level soil samples are analyzed against an aqueous (non-heated purge) calibration curve. The low level soils are analyzed against a calibration curve employing a heated purge temperature of 40°C.

4.10 Analysis sequence

IBLK (optional)
BFB
CCV
Method blank
Samples and QC

4.11 Sample Preparation

4.11.1 The samples are removed from the VOA refrigerator and are allowed to warm to room temperature. Aqueous samples which are received in 40 mL vials and low level soil samples containing the pre-weighed sample aliquot in sodium bisulfate are placed onto the autosampler carousel. See section 4.11.5 for the preparation of medium soils. The autosampler transfers a 5 mL or 25 mL aliquot of sample (or an aliquot of PFW for the soils) into the autosampler syringe, where it injects 1 µL of the internal standard/ surrogate standard solution.

4.11.2 The pH of all water and low level soil samples is measured subsequent to analysis in order to determine if they were preserved adequately without disturbing the integrity of the sample. The pH values when not preserved to a pH ≤2 are noted in the instrument logbook.

4.11.3 If the concentration of any target analyte exceeds the initial calibration range, the sample must be diluted and reanalyzed.

4.11.4 If aqueous samples require dilutions, they are diluted by one of two methods listed:

1) the samples are diluted with PFW in a volumetric where they are transferred to 40 mL vials and placed onto the autosampler, or

2) the undiluted samples are placed on the autosampler, where they are diluted with PFW by the autosampler. Refer to the instrumentation manuals for the complete operation of the autosampler.

4.11.5 Medium Soils

4.11.6 The soil sample is gently mixed (no supernatant liquid is removed) and a 5 gram aliquot (weighed out to the nearest 0.1 g) is transferred to a tared 15 mL vial. 10.0 mL of methanol is added to the vial. The vial is sealed, then shaken for two minutes. A 1 mL aliquot is transferred to a GC vial for storage in the dark at 4°C (±2°C). A 100 µL sample aliquot per 5 mL of PFW is placed onto the autosampler carousel. The autosampler transfers a volume of 5 mL of this sample into the autosampler syringe, where it injects 1 µL of the internal standard/ surrogate solution.

4.12 Qualitative Identification

4.12.1 Two criteria must be satisfied to verify the identification of target compounds:

1) elution of the sample component within 0.50 minutes of the standard component and,

2) appropriate correspondence between the sample mass spectrum and the standard mass spectra.

a) The three ions of greatest relative intensity, or any ions over 30% relative intensity which are present in the standard mass spectrum must be present in the sample spectrum.

b) The relative intensities of ions must be within 30% between the standard and sample spectra.

d) The emphasis of spectral review is on the three major ions. However, if a target compound cannot be verified by all the criteria listed above, but if, in the technical judgment of the analyst, the identification is correct, then the analyte will be quantitated, and reported. Compounds meeting the identification criteria must be reported with their spectra.

4.12.2 A library search may be performed for the purpose of identifying up to 10 largest non-target analytes also referred to as Tentatively Identified Compounds (TICs) if requested by the client. The criteria for confirming TICs are listed below.

1) Relative intensities of ions in the reference spectrum > 10% of the most abundant ion should be present in the sample spectrum.

2) Molecular ions in the reference spectrum should be present in the sample spectrum.

3) The relative intensities for the major ions should agree within 20%.

4) Ions in the sample spectrum but not present in the standard spectrum should be reviewed for possible background contamination or coelution.

6) If, after careful review and in the technical judgment of the analyst, no valid tentative identification can be made, the compound should be reported as follows:

A) If the library search produces a match at or above 85%, report that compound.
B) If the library search produces more than one compound at or above 85%, report the first compound.
C)If the library search produces no matches at or above 85%, report the compound as unknown. The analyst should give additional classification of the unknown compound, if possible (i.e., unknown hydrocarbon, unknown acid type, unknown chlorinated compound).

4.13 Manual Peak Integrations

4.13.1 Manual peak integrations should be used only when necessary to correct for matrix interference, tangent peaks, split peaks, overloaded peaks and rising baselines. Manual integrations are not to be used in an attempt to meet calibration, surrogate recovery, or spike recovery criteria. Refer to LTL-1020, *The Integration of IC, GC, HPLC, and GC/MS Peaks* for more detail.

4.13.2 If a manual integration is necessary, follow the following guidelines:

1) Integrate only the peak. Start where the peak lifts from the baseline and end as soon as it returns to the baseline. Do not integrate extra baseline in an attempt to increase peak area.

2) Do not "peak shave". Do not cut off legitimate parts of the peak in order to reduce peak area.

3) In cases of tangent skims, do not increase or decrease peak areas or heights by skimming extra long baselines or drawing the baseline too low.

4) Always initial and date your manual integrations.

4.14 Compound Quantification

4.14.1 Target compound concentrations are calculated using the following equations:

Aqueous samples:

$$\text{Conc } (\mu g / L) = \frac{A_x \times I_s \times Df}{A_{is} \times ARF \times V_o}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average RF from the ambient temperature purge of the initial calibration.
V_o = Volume of water purged (mL).
Df = Dilution factor. Assume 1.0 for no dilution.

Low soil (on a dry-weight basis)

$$\text{Conc } (\mu g / kg) = \frac{A_x \times I_s}{A_{is} \times ARF \times W_s \times D}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average response factor from the heated purge of the initial calibration.
W_s = Weight of sample added to purge vessel (g).

$$D = \frac{100 - \%\text{Moisture}}{100}$$

Medium soil (on a dry weight basis)

$$\text{Conc } (\mu g / kg) = \frac{A_x \times I_s \times V_t \times 1000 \times Df}{A_{is} \times ARF \times V_a \times W_s \times D}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average RF from the ambient temperature purge of the calibration standard.
V_t = Total volume of the methanol extract in milliliters (10 mL).
V_a = Volume of the methanol extract in microliters added to the reagent water for purging.
W_s = Weight of sample added to purge vessel (g).
Df = Dilution factor. Assume 1.0 for no dilution.

5. Reports

5.1 Data Packet Organization

5.1.1 See Appendix II for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and a MS/MSD report.

5.3 Control Limits

5.3.1 The laboratory has a computerized database which is used to generate control limits for various analyses, matrices, and analytes. The control limits define the range in which 99% of all values produced by a system should lie. Because the control limits are updated periodically, the current control limits for QC analyses have not been defined in this SOP. A copy of all current control limits are located in Laucks' Control Limits Database.

5.4 Data Qualifying Flags

5.4.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the limit of detection indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
N	Used to denoted the presumptive evidence of a target analyte. This flag is used when it is not possible to confirm the presence of a compound by the strict guidelines for mass spectral interpretation, but in the technical judgment of the analyst, this compound is present.

6. Quality Control

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 Initial calibration data are evaluated using %RSD and the relative response factors.

RFs are calculated using the equation:

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_s}$$

where:

A_x = Response of target analyte
A_{is} = Response of internal standard
C_x = Amount of target analyte (ng/mL).
C_{is} = Amount of internal standard (ng/mL).

%RSDs are calculated using the equation:

$$\%RSD = \frac{SD_i}{RF_i} \times 100$$

where:

Sd_i = Standard deviation of the initial calibration points
RF_i = Average RF from initial calibration

6.1.2 The System Performance Check Compounds (SPCCs) are checked to determine that the minimum average relative response factors tabulated below are met, and that the maximum RSD for each Calibration Check Compound (CCC) is ≤ 30%. The RSD for all other target analytes *should* be ≤ 15%. If the RSD of any target analyte is greater than or equal to 15%, but ≤ 30%, additional calibration options may be used. Alternatively, if the mean of the RSD values for all analytes in the calibration is ≤ 15%, the initial calibration is considered acceptable. Laucks uses 40% percent as the upper limit for all compounds as an internal guideline although it is not a requirement of SW 846.

6.1.2.1 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.

6.1.2.2 The data used must be supplied with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded 15% and the results of the mean RSD calculation.

SPCC Compounds	Minimum RF Limit
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
Chlorobenzene	0.300
1,1,2,2-Tetrachloroethane	0.300

CCC Compounds	%RSD Limit
Vinyl chloride	≤30%
1,1-Dichloroethene	≤30%
Chloroform	≤30%
1,2-Dichloropropane	≤30%
Toluene	≤30%
Ethylbenzene	≤30%

6.1.3 Corrective action

6.1.3.1 If the criteria are not met, additional standards may be analyzed or appropriate instrument maintenance and analysis of new standards should be performed.

6.1.4 Documentation

6.1.4.1 Copies of the initial calibration standards and the calculated RRFs and RSDs are stored with the raw data. If criteria are not met, reanalysis of additional standard(s) are noted in the instrument run log.

6.2 Continuing Calibration Verification

6.2.1 Criteria

6.2.1.1 At the beginning of each 12 hour QC period, a CCV standard is analyzed. The RRF for each compound is calculated and the percent difference is calculated as follows:

$$\%D = \frac{RF_i - RF_c}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration
RF_c = RF from CCV standard

6.2.1.2 The minimum RRFs for the SPCCs must meet the criteria applied in the initial calibration detailed above. The %D for the CCCs cannot exceed the 20% CCV criteria as tabulated below.

CCC Compounds	% D Limit
1,1-Dichloroethene	≤20%
Chloroform	≤20%
1,2-Dichloropropane	≤20%
Toluene	≤20%
Ethylbenzene	≤20%
Vinyl chloride	≤20%

6.2.1.3 Laucks uses a maximum %D of 40% for all other reported analytes as an internal guideline, although it is not a requirement of SW 846.

6.2.2 Corrective action

6.2.2.1 Check calculations, check standard solutions or perform instrument maintenance. Reanalyze the standard, if this fails, re-do initial calibration.

6.2.3 Documentation

6.2.3.1 Copies of the continuing calibration standards and the calculated RFs and %Ds are stored with the raw data. If criteria are not met, reanalysis of additional standard(s) are noted in the instrument run log.

6.3 Instrument Blank

6.3.1 Criteria

6.3.1.1 The criteria used are the same as those used for the method blank control limits.

6.3.2 Corrective action

6.3.2.1 If the initial IBLK contains measurable levels of target analytes above one-half the reporting limit, except where noted below, the system is out of control. The source of contamination must be identified and corrected. If analysis of an additional IBLK does not demonstrate that the analytical system is free of contaminants, then instrument maintenance such as cleaning the transfer lines or replacing the trap may be required.

6.3.3 Documentation

6.3.3.1 Copies of instrument blank analyses are stored with the sample analyses raw data. Re-analysis of the method blank and/or maintenance performed is documented in the instrument run log.

6.4 Method Blanks

6.4.1 Criteria

6.4.1.1 A method blank is used to verify contamination free reagents and apparatus. A method blank is analyzed following every CCV and prior to sample analysis. Method blank criteria are listed below.

6.4.1.2 Laucks criteria for a method blank is less than one-half the RL for each analyte.

6.4.2 Corrective action

6.4.2.1 When these criteria are exceeded, corrective action should be taken to find/reduce/eliminate the source of this contamination in the method blank. Sample corrective action may be limited to qualification for blank contamination. When the concentrations of any target analytes within the method blank are above one-half of the reporting limit, the effect this may have had on the samples will be assessed as detailed below:

1) If any analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps shall be taken to

find/reduce/eliminate the source of this contamination in the method blank. The case narrative should also discuss the situation.

2) If an analyte is found in the method blank and some, or all, of the other batch samples, additional corrective action is required to reanalyze the method blank, and any samples containing the same contaminant(s) which exceeded the corrective action limit(s). If the contamination remains, the contaminated samples of the batch would be reprepared and reanalyzed with a new method blank and batch specific QC samples.

3) Sporadic cases of contamination may be difficult to control however, daily contamination would not be acceptable.

4) Out-of-control surrogate recoveries in the method blank require reanalysis of the method blank.

5) If common common lab contaminates; such as acetone and methylene chloride are present, up to the reporting limit, the assoicated sample results for these compounds are flagged appropriately. Analytes may be present as long as they are less than 10 percent of the regulatory limit associated with that analyte, or less than 10 percent of the sample result for the same analyte, whichever is greater for the method blank to be acceptable. In addition, bromomethane may be present up to the RL in a medium soil method blank. This is due to the foaming action of the methanol with the sparging frit and is very difficult to control.

6.4.3 Documentation

6.4.3.1 Copies of all method blank analyses are stored with the sample analyses raw data. In addition, the raw data for the method blank analyses are stored in the laboratory for an extended period of time. The results for the method blank analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank(s) are noted in the instrument run log. Outliers are discussed in the case narrative.

6.5 Blank Spikes

6.5.1 Criteria

6.5.1.1 A blank spike follows the same protocol as a matrix spike analysis except that the spiking solution is added to an aliquot of PFW (or sand for soil samples) instead of an actual sample. A method blank with added analytes is a blank spike. A blank spike is the

same as a QC check standard. It is recommended that all target analytes of concern be spiked into the LCS. A subset of the single-component target analytes can be substituted for the full list if specified in project-specific contracts or workplans per the USACOE Shell document, or if the target list contains more than 50 components per the DOD Quality Systems Manual- Final Version 1.

6.5.1.2 A blank spike may be used to fill the requirements for the performance of an initial calibration and continuing calibration verification standard. In addition, the level spiked will periodically be varied within the established calibration range.

6.5.2 Corrective action

6.5.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample reanalysis would be triggered by an out of control blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

6.5.3 Documentation

6.5.3.1 The raw data for the blank spike analyses are stored in the laboratory for an extended period of time. The results for the blank spike analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank spike(s) are noted in the instrument run log.

6.6 Matrix Spike

6.6.1 Criteria

6.6.1.1 A sample is chosen at random (unless specified by a client) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to analysis. A matrix spike analysis is performed with each analytical batch, up to a maximum of 20 samples. The matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$Recovery = \frac{SS - S}{SpikedAmount} \times 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample

6.6.2 Corrective action

6.6.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation and/or reanalysis. This process should also include evaluation of the recovery of surrogate compounds in the MS sample and recovery of matrix spiking compounds from the blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.6.3 Documentation

6.6.3.1 The raw data for the matrix spike analyses are stored with the sample analyses raw data. The results for the matrix spike analyses are reported electronically via the laboratory's LIMS database.

6.7 Matrix Spike Duplicate

6.7.1 Criteria

6.7.1.1 A matrix spike duplicate analysis is performed with each analytical batch, up to a maximum of 20 samples. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method

precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2) \backslash 2} \times 100$$

where:

S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

6.7.2 Corrective action

6.7.2.1 If a trend in out-of-control RPD values is observed, the method used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.7.3 Documentation

6.7.3.1 The raw data for the matrix spike duplicate analyses are stored with the sample analyses raw data. The results for the matrix spike duplicate analyses are reported electronically via the laboratory's LIMS database. Any out-of-control results are documented in the LIMS database and/or analytical narrative.

6.8 Surrogate Recovery

6.8.1 Criteria

6.8.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follow:

$$\text{Recovery} = \frac{S_m}{S_a} \times 100$$

where:

S_m = concentration of surrogate measured in sample
S_a = concentration of surrogate added

6.8.2 Corrective Action

6.8.2.1 Check calculations for possible error. Reanalysis is required for all surrogate recoveries which exceed the established control limits.

6.8.2.2 Out-of-control surrogate recoveries in the method blank require that the method blank be reanalyzed. Sample analyses are not performed until all criteria are met in the method blank analysis. Sample and QC analyses require reanalysis for out-of-control surrogate recoveries. If the sample reanalyses also demonstrate out-of-control surrogate recoveries due to matrix interference, this corrective action is discussed in the narrative.

6.8.3 Documentation

6.8.3.1 The surrogate recoveries for all samples, blanks, and QC analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis for out-of-control surrogate recoveries are designated by an "RE" in the instrument run log.

7. References

1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 8260B, 3rd edition, 2nd revision, December 1996.

2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 8000B, 3rd edition, 2nd revision, December 1996.

3) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5000B, 3rd edition, revision 0, December 1996.

4) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5030B, 3rd edition. 2nd revision, December 1996.

5) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5035, 3rd edition, revision 0, December 1996.

APPENDIX I

Initial Calibration Concentrations For Low Level (25 mL) Waters (µg/L)

Compound	STD1	STD2	STD3	STD4	STD5
Dichlorodifluoromethane	1	5	10	25	50
Chloromethane	1	5	10	25	50
Vinyl Chloride	1	5	10	25	50
Bromomethane	1	5	10	25	50
Chloroethane	1	5	10	25	50
Trichlorofluoromethane	1	5	10	25	50
1,1-Dichloroethene	1	5	10	25	50
Acetone	5	10	20	50	100
Carbon disulfide	1	5	10	25	50
Methylene chloride	1	5	10	25	50
trans-1,2-Dichloroethene	1	5	10	25	50
1,1-Dichloroethane	1	5	10	25	50
Vinyl acetate	1	5	10	25	50
2,2-Dichloropropane	1	5	10	25	50
cis-1,2-Dichloroethene	1	5	10	25	50
2-Butanone	5	10	20	50	100
Bromochloromethane	1	5	10	25	50
Chloroform	1	5	10	25	50
1,1,1-Trichloroethane	1	5	10	25	50
Carbon tetrachloride	1	5	10	25	50
1,1-Dichloropropene	1	5	10	25	50
Benzene	1	5	10	25	50
1,2-Dichloroethane	1	5	10	25	50
Trichloroethene	1	5	10	25	50
1,2-Dichloropropane	1	5	10	25	50
Dibromomethane	1	5	10	25	50
Bromodichloromethane	1	5	10	25	50
cis-1,3-Dichloropropene	1	5	10	25	50
4-Methyl-2-pentanone	5	10	20	50	100
Toluene	1	5	10	25	50
trans-1,3-Dichloropropene	1	5	10	25	50

Compound	STD1	STD2	STD3	STD4	STD5
1,1,2-Trichloroethane	1	5	10	25	50
Tetrachloroethene	1	5	10	25	50
1,3-Dichloropropane	1	5	10	25	50
2-Hexanone	5	10	20	50	100
Chlorodibromomethane	1	5	10	25	50
1,2-Dibromoethane	1	5	10	25	50
Chlorobenzene	1	5	10	25	50
1,1,1,2-Tetrachloroethane	1	5	10	25	50
Ethylbenzene	1	5	10	25	50
m,p-Xylene	2	10	20	50	100
o-Xylene	1	5	10	25	50
Styrene	1	5	10	25	50
Bromoform	1	5	10	25	50
Isopropylbenzene	1	5	10	25	50
Bromobenzene	1	5	10	25	50
1,2,3-Trichloropropane	1	5	10	25	50
1,1,2,2-Tetrachloroethane	1	5	10	25	50
n-Propylbenzene	1	5	10	25	50
2-Chlorotoluene	1	5	10	25	50
4-Chlorotoluene	1	5	10	25	50
1,3,5-Trimethylbenzene	1	5	10	25	50
tert-Butylbenzene	1	5	10	25	50
1,2,4-Trimethylbenzene	1	5	10	25	50
sec-Butylbenzene	1	5	10	25	50
1,3-Dichlorobenzene	1	5	10	25	50
1,4-Dichlorobenzene	1	5	10	25	50
p-Isopropyltoluene	1	5	10	25	50
1,2-Dichlorobenzene	1	5	10	25	50
n-Butylbenzene	1	5	10	25	50
1,2-Dibromo-3-chloropropane	1	5	10	25	50
1,2,4-Trichlorobenzene	1	5	10	25	50
Naphthalene	1	5	10	25	50
Hexachlorobutadiene	1	5	10	25	50
1,2,3-Trichlorobenzene	1	5	10	25	50
Methyl tert-butyl ether	1	5	10	25	50

Initial Calibration Concentrations Of Routine Target Analytes For Low And Medium Level Soils & 5 mL Waters

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5
Dichlorodifluoromethane	3	10	50	100	200
Chloromethane	3	10	50	100	200
Vinyl Chloride	3	10	50	100	200
Bromomethane	3	10	50	100	200
Chloroethane	3	10	50	100	200
Trichlorofluoromethane	3	10	50	100	200
1,1-Dichloroethene	3	10	50	100	200
Acetone	10	20	50	100	200
Carbon disulfide	3	10	50	100	200
Methylene chloride	3	10	50	100	200
trans-1,2-Dichloroethene	3	10	50	100	200
1,1-Dichloroethane	3	10	50	100	200
cis-1,2-Dichloroethene	3	10	50	100	200
2-Butanone	10	20	50	100	200
Chloroform	3	10	50	100	200
1,1,1-Trichloroethane	3	10	50	100	200
Carbon tetrachloride	3	10	50	100	200
Benzene	3	10	50	100	200
1,2-Dichloroethane	3	10	50	100	200
Trichloroethene	3	10	50	100	200
1,2-Dichloropropane	3	10	50	100	200
Bromodichloromethane	3	10	50	100	200
cis-1,3-Dichloropropene	3	10	50	100	200
4-Methyl-2-pentanone	10	20	50	100	200
Toluene	3	10	50	100	200
trans-1,3-Dichloropropene	3	10	50	100	200
1,1,2-Trichloroethane	3	10	50	100	200
Tetrachloroethene	3	10	50	100	200
2-Hexanone	10	20	50	100	200
Chlorodibromomethane	3	10	50	100	200
Chlorobenzene	3	10	50	100	200
Ethylbenzene	3	10	50	100	200
m,p-Xylene	6	20	100	200	400
o-Xylene	3	10	50	100	200
Styrene	3	10	50	100	200
Bromoform	3	10	50	100	200

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5
1,1,2,2-Tetrachloroethane	3	10	50	100	200

Initial Calibration Concentrations Of Additional Analytes For Low And Medium Level Soils & 5 mL Waters

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5
1,1,1,2-Tetrachloroethane	3	10	50	100	200
2,2-Dichloropropane	3	10	50	100	200
Bromochloromethane	3	10	50	100	200
1,1-Dichloropropene	3	10	50	100	200
1,2-Dibromoethane	3	10	50	100	200
Dibromomethane	3	10	50	100	200
1,3-Dichloropropane	3	10	50	100	200
Isopropylbenzene	3	10	50	100	200
Bromobenzene	3	10	50	100	200
1,2,3-Trichloropropane	3	10	50	100	200
n-Propylbenzene	3	10	50	100	200
2-Chlorotoluene	3	10	50	100	200
4-Chlorotoluene	3	10	50	100	200
1,3,5-Trimethylbenzene	3	10	50	100	200
tert-Butylbenzene	3	10	50	100	200
1,2,4-Trimethylbenzene	3	10	50	100	200
sec-Butylbenzene	3	10	50	100	200
1,3-Dichlorobenzene	3	10	50	100	200
1,4-Dichlorobenzene	3	10	50	100	200
p-Isopropyltoluene	3	10	50	100	200
1,2-Dichlorobenzene	3	10	50	100	200
n-Butylbenzene	3	10	50	100	200
1,2-Dibromo-3-chloropropane	3	10	50	100	200
1,2,4-Trichlorobenzene	3	10	50	100	200
Naphthalene	3	10	50	100	200
Hexachlorobutadiene	3	10	50	100	200
1,2,3-Trichlorobenzene	3	10	50	100	200
Iodomethane	3	10	50	100	200
trans-1,4-Dichloro-2-butene	3	10	50	100	200
Methyl tert-butyl ether	3	10	50	100	200

APPENDIX II

Data Packet Sequence

1. QC Summary
   - Surrogate recoveries
   - MS/MSD summary
   - Method blank summary
   - Instrument performance check (tuning)
   - Internal standard areas and RT summary

2. Sample Data
   - Target compound results
   - TICs (if requested)
   - Chromatogram normalized to nonsolvent peak
   - Quantitation report
   - Spectra

3. Standards Data
   - Initial calibration form
   - Chromatograms
   - Quantitation reports

   - CCV form
   - Chromatograms
   - Quantitation reports

4. Raw QC
   - BFB tune spectrum, chromatogram, and tabular listing
   - Method blank results, TICs, chromatogram, quantitation report, and spectra
   - MS/MSD results, chromatogram, quantitation report, spectra and control charts

5. Bench Sheets
   - Copy of instrument logbook, copy of COC, copy of holding blank analysis

6. Narrative.

7. Electronic Disk Deliverables (EDD) when requested

8. Control charts

APPENDIX III

Determination of Vinyl Chloride by Selected Ion Monitoring (SIM)

Analytical Conditions:

GC Parameters

Injection Port Temperature	: 200°C
GC/MS Interface Temperature	: 250°C
Initial GC Temperature	: 30°C
Initial Hold Time	: 0 min
Ramp Rate	: 6°C/min
Final Temperature	: 105°C
Final Time	: 0 min
Carrier Gas Flow	: 15 mL/min
Column Head Pressure	: 9 psi

Purge and Trap Parameters

Purge Time	: 11 min
Desorb Time	: 1 min
Bake Time	: 16 min
Desorb Preheat Temperature	: 20°C
Desorb Temperature	: 180°C
Purge Flow	: 40 mL/min
Purge Temperature	: Ambient
Desorb Flow Rate	: 15 mL/min

Mass Spectrometer Parameters

Electron Energy	: 70 volts
Acquisition Start Time	: 0.1 min

Group 1:

M/Z	Dwell Time (milliseconds)
61	50
62	400
63	50
64	50

Group 2:

M/Z	Dwell Time (milliseconds)
49	50
51	50
128	100
129	50

Group 3:

M/Z	Dwell Time (milliseconds)
65	50
67	50
102	50
104	50

Standards:

Internal Standard: Bromochloromethane
Surrogate: 1,2-Dichloroethane-d4

APPENDIX IV

Additional Appendix IX Compounds And Their Initial Concentrations For Low Level (25mL) Waters

Compound* (µg/L)	STD1	STD2	STD3	STD4	STD5
Acrolein	10	20	50	100	200
Bromoethane	10	20	50	100	200
Allyl chloride	10	20	50	100	200
Acrylonitrile	10	20	50	100	200
Chloroprene	10	20	50	100	200
Methacrylonitrile	10	20	50	100	200
Ethyl methacrylate	10	20	50	100	200
trans-1,4-Dichloro-2-butene	10	20	50	100	200
2-Chloroethylvinyl ether[1]	10	20	50	100	200

* The calibration range defines the reporting range for these compounds. However, the reporting limits may change for individual projects, based on MDLs, mass spectral confirmation, or project-specific requirements.

1 This analyte is not recommended because it decomposes in water, and does not recover in acidified samples.

APPENDIX V

Method 8260 Requirements and Corrective Actions

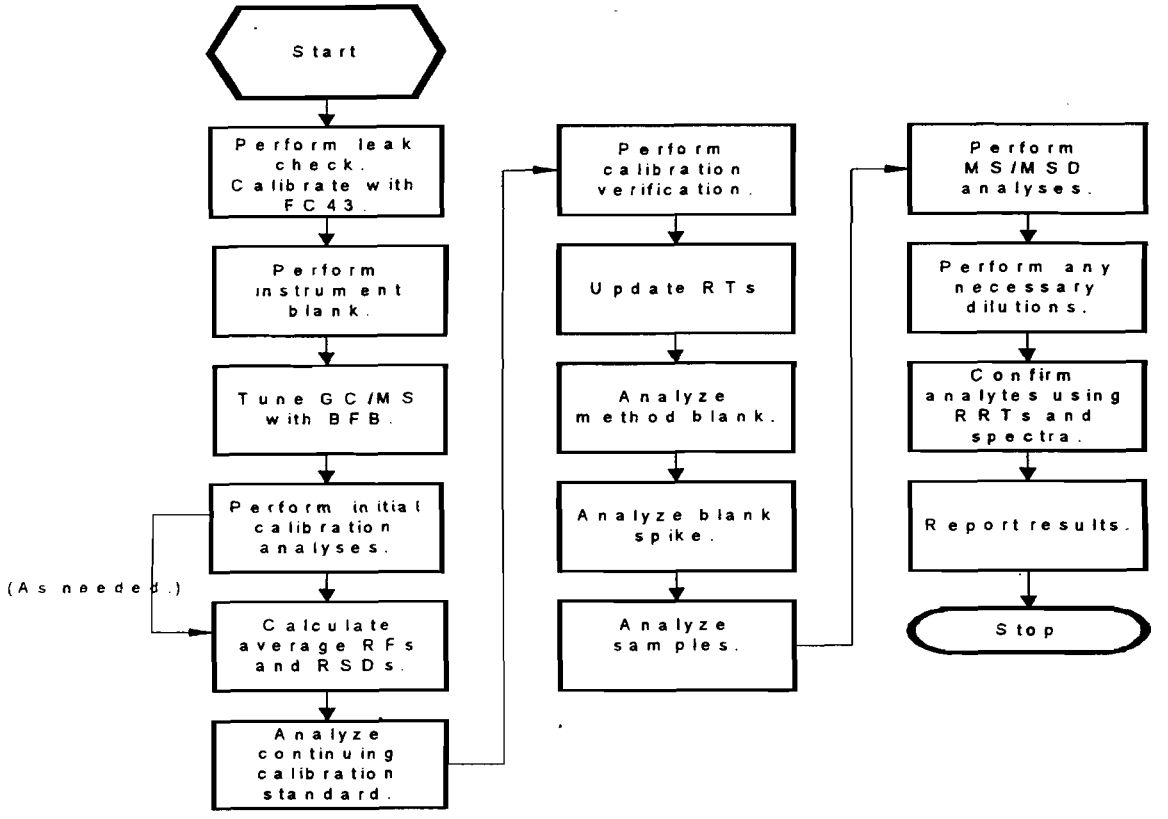
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Holding Time	14 days from collection.	14 days from collection.	N/A	N/A	HTVR (holding time violation report).
Tuning Verification 5 - 50 ng BFB	Mass Abundance 50 15-40% of 95 75 30-60% of 95 95 100% 96 5-9% of 95 173 < 2% of 174 174 > 50% of 95 175 5-9% of 174 176 95-101% of 174 177 5-9% of 176 *Alternate tuning criteria may be used (e.g.,CLP,524.2).	Mass Abundance 50 8-40% of 95 75 30-66% of 95 95 100% 96 5-9% of 95 173 < 2% of 174 174 50-120% of 95 175 4-9% of 174 176 93-101% of 174 177 5-9% of 176 * Alternate tuning criteria may be used.	Every 12 hours.	Re-tune instrument to meet criteria.	Copy of BFB raw data and tune criteria with file.
Initial Calibration	Minimum of 5 levels with lowest near RL. %RSD for CCCs ≤30% RF for SPCCs: chloromethane 0.10 1,1-dichloroethane 0.10 bromoform 0.10 chlorobenzene 0.30 1,1,2,2-tetrachloroethane 0.30 Non-CCCs: all analytes or average of all analytes = ≤15%.	5 levels 1, 5, 10, 20, 50 µg/L. (Ketones 5,10,20,50,100 µg/L) %RSD for CCCs ≤30% RFs for SPCCs: chloromethane 0.10 1,1-dichloroethane 0.10 bromoform 0.10 chlorobenzene 0.30 1,1,2,2-tetrachloroethane 0.30 Non-CCCs: all analytes or average of all analytes ≤15%. 40%RSD for each analyte as guideline.	Initially.	Reanalysis of out of control standards.	Copies of all raw data, tune file, and Form VI.
Initial Calibration Verification		Typically mid-range standard. Must meet limits applicable to CCV.	Once after initial calibration.	Re-analysis of initial calibration.	
Continuing Calibration Verification	Mid-level calibration standard every 12 hours. RF for SPCCs same as initial. RF for CCCs must be ≤20% D.	%D for CCC ≤20% %D for all other compounds < 40% as laboratory guideline.	Every 12 hours.	Reanalyze for analytes that exceed the control limits.	Copies of raw data and Form VII.
Internal Standards	Fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, recommended.	Fluorobenzene, chlorobenzene-d5 and 1,4-dichlorobenzene-d4. RT must be ± 30 seconds from last calibration; area must be 50 to 100%.	Every sample.	Reanalyze all samples that do not meet these criteria.	IS areas and RTs are submitted on Form 8, and included in data package.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank/ Instrument Blank	One method blank per extraction batch (up to 20 samples) or when there is a change in reagents, whichever is more frequent.	< 1/2 Reporting limit.	One method blank per QC period, following the CCV, prior to sample analysis. Typically, an instrument blank is analyzed prior to tuning the instrument.	Evaluate, qualify.	Copies of raw data filed, and reported electronically. Daily control charts for all method blanks are recorded. Narrate any analytes >1/2 RL
Surrogate Standards	4-bromofluorobenzene, dibromofluoromethane 1,2-dichloroethane-d4 or toluene-d8 recommended. Compare %R to laboratory established limits.	1,2-dichloroethane-d4, toluene-d8, 4-bromofluorobenzene, and dibromofluoromethane. Percent recoveries must meet established laboratory control limits. See QC database for control limits.	Every sample.	Reanalysis if surrogate recoveries exceed the established control limits.	Surrogates that exceed the control limits are documented in associated file.
Quality Control	One MS/MSD per 20 samples or each batch of samples, whichever is more frequent. Compare % recovery to laboratory established limits.	Blank spike recoveries and RPDs must meet control limits established by laboratory. MS/MSD recoveries are allowed to be out if due to matrix effect. See QC database for control limits.	One MS/MSD per 20 samples. A blank spike is analyzed every QC period.	Reanalysis of MS/MSD unless matrix effect has already been demonstrated. This can be determined by comparing with the blank spike recoveries.	Blank spike and MS/MSD recoveries are stored electronically. Raw data is stored with file. Out of control events are notated in file.
Qualitative Identification	All ions > 10% intensity must be ± 20% of standard; ± 0.06 RRT units of standard RRT.	All ions > 10% intensity must be ± 20% of standard; ± 0.50 min. of standard RT.	For identifying all target analytes in each sample.		Copies of all mass spectra stored with data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Sample Preservation and Storage	pH ≤2 with HCL for waters. pH ≤ 2 with sodium bisulfate for low level soils. Medium soils: zero headspace, methanol, or EnCore™ sampler. Store at 4 ° C.	pH ≤2 with HCL for waters. pH ≤2 with sodium bisulfate for low level soils. Medium soils: zero headspace, methanol, or EnCore™ sampler. Store at 4° C.	All water and low level soil samples.	If the water and low level soil samples are not acid-preserved, this is narrated in final report.	pH of all water samples and low level soils, where not preserved to a pH of ≤2, noted in instrument logbook.
Standard Solution Expiration	Stock standards (except gases): 6 months. Store between -10 to -20° C in teflon-sealed liners and amber bottles with minimal headspace, protect from light. Stock gas standards: 2 months. Storage conditions same as stock standards. Calibration standards: daily.	Stock standards (except gases): 6 months. Gas stock standards: 2 months. Stored at -10° to -20° C . Calibration standards: daily. All standards sealed in flame-sealed ampules: 2 years (not to exceed parent expiration date).			All standard data recorded in standard logbook.
Field Sample Amount	2 40 mL vials with zero headspace. Soils: vials, with zero headspace, or a sample aliquot in 40 mL vial. Glass container, septum-sealed screw-caps.	Waters:4 40 mL vials with zero headspace. Soils: 2 oz vials, with zero headspace, or a sample aliquot in 4 -40 mL vial. Glass container, septum-sealed screw-caps.			Bottles received by laboratory documen electronically.
Applicability	Groundwaters, soils, sediments, sludges, non-water miscible wastes and others.				

APPENDIX VI

Method 8260B Flow Chart



Refer to correctve action chart for out of control events.

APPENDIX VII

Method 8260 Target Analytes

Dichlorodifluoromethane	Chloromethane
Vinyl Chloride	Bromomethane
Chloroethane	Trichlorofluoromethane
1,1-Dichloroethene	Acetone
Carbon disulfide	Methylene chloride
trans-1,2-Dichloroethene	1,1-Dichloroethane
1,1,2,2-Tetrachloroethane	2,2-Dichloropropane
cis-1,2-Dichloroethene	2-Butanone
Bromochloromethane	Chloroform
1,1,1-Trichloroethane	Carbon tetrachloride
1,1-Dichloropropene	Benzene
1,2-Dichloroethane	Trichloroethene
1,2-Dichloropropane	Dibromomethane
Bromodichloromethane	cis-1,3-Dichloropropene
4-Methyl-2-pentanone	Toluene
trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Tetrachloroethene	1,3-Dichloropropane
2-Hexanone	Chlorodibromomethane
1,2-Dibromoethane	Chlorobenzene
1,1,1,2-Tetrachloroethane	Ethylbenzene
m,p-Xylene	o-Xylene
Styrene	Bromoform
Methyl tert-butyl ether	

APPENDIX VIII

Method 8260 Optional Analytes for Low Level (25 mL) Water Analyses

Isopropylbenzene	Bromobenzene
1,2,3-Trichloropropane	2-Chlorotoluene
n-Propylbenzene	1,3,5-Trimethylbenzene
4-Chlorotoluene	1,2,4-Trimethylbenzene
tert-Butylbenzene	1,3-Dichlorobenzene
sec-Butylbenzene	p-Isopropyltoluene

Method 8260 Optional Analytes for Low Level (25 mL) Water Analyses

1,4-Dichlorobenzene	n-Butylbenzene
1,2-Dichlorobenzene	1,2,4-Trichlorobenzene
1,2-Dibromo-3-chloropropane	Hexachlorobutadiene
Naphthalene	1,2,3-Trichlorobenzene
Vinyl acetate	

Appendix IX

Additional Appendix IX Compounds (25 mL)

Acrolein	Bromoethane
Allyl chloride	Acrylonitrile
Chloroprene	Methacrylonitrile
Methyl methacrylate	trans-1,4-Dichloro-2-butene
2-Chloroethylvinyl ether	Ethyl methacrylate
Iodomethane	

APPENDIX X

Target Analytes And Their Associated Internal Standards

Fluorobenzene	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**
Dichlorodifluoromethane	Toluene	Isopropylbenzene
Chloromethane	trans-1,3-Dichloropropene	Bromobenzene
Vinyl Chloride	1,1,2-Trichloroethane	1,2,3-Trichloropropane
Bromomethane	Tetrachloroethene	1,1,2,2-Tetrachloroethane
Chloroethane	1,3-Dichloropropane	n-Propylbenzene
Trichlorofluoromethane	2-Hexanone	2-Chlorotoluene
1,1-Dichloroethene	Chlorodibromomethane	4-Chlorotoluene
Acetone	1,2-Dibromoethane	1,3,5-Trimethylbenzene
Carbon disulfide	Chlorobenzene	tert-Butylbenzene
Methylene chloride	1,1,1,2-Tetrachloroethane	1,2,4-Trimethylbenzene
trans-1,2-Dichloroethene	Ethylbenzene	sec-Butylbenzene
1,1-Dichloroethane	m,p-Xylene	1,3-Dichlorobenzene
Vinyl acetate	o-Xylene	1,4-Dichlorobenzene

Fluorobenzene	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**
2,2-Dichloropropane	Styrene	p-Isopropyltoluene
cis-1,2-Dichloroethene	Bromoform	1,2-Dichlorobenzene
2-Butanone		n-Butylbenzene
Bromochloromethane		1,2-Dibromo-3-chloropropane
Chloroform		1,2,4-Trichlorobenzene
1,1,1-Trichloroethane		Naphthalene
Carbon tetrachloride		Hexachlorobutadiene
1,1-Dichloropropene		1,2,3-Trichlorobenzene
Benzene		
1,2-Dichloroethane		
Trichloroethene		
1,2-Dichloropropane		
Dibromomethane		
Bromodichloromethane		
cis-1,3-Dichloropropene		
4-Methyl-2-pentanone		
Methyl tert-butyl ether		

Additional Appendix IX Compounds And Associated Internal Standard

Fluorobenzene (IS)	**Chlorobenzene-d5**
Acrolein	Ethyl methacrylate
Bromoethane	
Allyl chloride	
Acrylonitrile	
Chloroprene	
Methacrylonitrile	
Methyl methacrylate	
trans-1,4-Dichloro-2-butene	
Iodomethane	
2-Chloroethylvinyl ether	

APPENDIX XI

Target Analytes And Their Quantitation Ions

Compound	Primary Quant Ions*	Secondary Quant Ions*
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	96	94
Chloroethane	64	66
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61,63
Acetone	43	58
Carbon disulfide	76	78
Methylene chloride	84	86
trans-1,2-Dichloroethene	96	98,61
1,1-Dichloroethane	63	65,83
2,2-Dichloropropane	77	97
cis-1,2-Dichloroethene	96	98,61
2-Butanone	43	57
Bromochloromethane	128	49,130
Chloroform	83	85
1,1,1-Trichloroethane	97	99,61
Carbon tetrachloride	117	119
1,1-Dichloropropene	75	77,110
Benzene	78	-
1,2-Dichloroethane	62	98
Trichloroethene	95	97,130,132
1,2-Dichloropropane	63	112
Dibromomethane	93	95,174
Bromodichloromethane	83	85,127
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58,85
Toluene	92	91
trans-1,3-Dichloropropene	75	77

Compound	Primary Quant Ions*	Secondary Quant Ions*
1,1,2-Trichloroethane	83	97,85
Tetrachloroethene	166	164
1,3-Dichloropropane	76	78
2-Hexanone	43	58,100
Chlorodibromomethane	129	208,206
1,2-Dibromoethane	107	109
Chlorobenzene	112	114
1,1,1,2-Tetrachloroethane	131	133
Ethylbenzene	91	106
m,p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78
Bromoform	173	175
Isopropylbenzene	105	120
Bromobenzene	156	158
1,2,3-Trichloropropane	75	77
1,1,2,2-Tetrachloroethane	83	85
n-Propylbenzene	120	91
2-Chlorotoluene	126	91
4-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
tert-Butylbenzene	91	134,119
1,2,4-Trimethylbenzene	105	120
sec-Butylbenzene	105	134

Compound	Primary Quant Ions*	Secondary Quant Ions*
1,3-Dichlorobenzene	146	148,111
1,4-Dichlorobenzene	146	148,111
p-Isopropyltoluene	119	134,91
1,2-Dichlorobenzene	146	148,111
n-Butylbenzene	91	92
1,2-Dibromo-3-chloropropane	75	155,157
1,2,4-Trichlorobenzene	180	182
Naphthalene	128	-
Hexachlorobutadiene	225	223,227
1,2,3-Trichlorobenzene	180	182
Methyl tert-butyl ether	73	57,41

Additional Appendix IX Compounds And Their Quantitation Ions

Compound	Primary Quant Ions*	Secondary Quant Ions*
Acrolein	56	55,58
Bromoethane	108	-
Allyl chloride	41	78
Acrylonitrile	53	52,51
Chloroprene	53	51
Methacrylonitrile	41	39
Ethyl methacrylate	69	-
Methyl methacrylate	41	39
trans-1,4-Dichloro-2-butene	53	75
Vinyl acetate	43	86
Iodomethane	142	127
Vinyl acetate	43	86
2-Chloroethylvinyl ether	63	65

* Primary and secondary quantitation ions assigned by Laucks may be slightly different than those recommended in SW 846, in order to avoid interference.

APPENDIX XII

Target Analytes, Their Elution Order And Standard Mixes

Compound	Standard Mix
Dichlorodifluoromethane	6
Chloromethane	6
Vinyl Chloride	6
Bromomethane	6
Chloroethane	6
Trichlorofluoromethane	6
Acrolein	8
1,1-Dichloroethene	5
Acetone	7
Iodomethane	9
Bromoethane	10
Carbon disulfide	11
Allyl Chloride	12
Methylene chloride	5
trans-1,2-Dichloroethene	5
Acrylonitrile	13
1,1-Dichloroethane	4
Vinyl acetate	14
Chloroprene	15
2,2-Dichloropropane	4
cis-1,2-Dichloroethene	5
2-Butanone	7
Methacrylonitrile	12
Bromochloromethane	4
Chloroform	4
1,1,1-Trichloroethane	4
Carbon tetrachloride	4
1,1-Dichloropropene	3
1,2-Dichloroethane-d4 (surr)	18
Benzene	2
1,2-Dichloroethane	3
Trichloroethene	3
1,2-Dichloropropane	3
Dibromomethane	4
Methyl methacrylate	12

Compound	Standard Mix
Bromodichloromethane	5
2-Chloroethylvinyl ether	16
cis-1,3-Dichloropropene	3
Ethyl methacrylate	12
4-Methyl-2-pentanone	7
Toluene-d8 (surr)	18
Toluene	2
trans-1,3-Dichloropropene	3
1,1,2-Trichloroethane	3
Tetrachloroethene	4
1,3-Dichloropropane	3
2-Hexanone	7
Chlorodibromomethane	5
1,2-Dibromoethane	3
Chlorobenzene	1
1,1,1,2-Tetrachloroethane	3
Ethylbenzene	2
m,p-Xylene	1,2
o-Xylene	1
Styrene	2
Bromoform	4
Isopropylbenzene	1
4-Bromofluorobenzene (surr)	18
Bromobenzene	2
1,2,3-Trichloropropane	3
1,1,2,2-Tetrachloroethane	3
n-Propylbenzene	1
trans-1,4-Dichloro-2-butene	17
2-Chlorotoluene	1
4-Chlorotoluene	1
1,3,5-Trimethylbenzene	2
tert-Butylbenzene	1
1,2,4-Trimethylbenzene	2

Compound	Standard Mix
sec-Butylbenzene	1
1,3-Dichlorobenzene	1
1,4-Dichlorobenzene	1
p-Isopropyltoluene	2
1,2-Dichlorobenzene	1
n-Butylbenzene	2
1,2-Dibromo-3-chloropropane	3
1,2,4-Trichlorobenzene	2
Naphthalene	2
Hexachlorobutadiene	3
1,2,3-Trichlorobenzene	2
Methyl tert-butyl ether	19

Key:

1 = Supelco VOC Mix 1
2 = Supelco VOC Mix 2
3 = Supelco VOC Mix 3
4 = Supelco VOC Mix 4
5 = Supelco VOC Mix 5
6 = Supelco VOC Mix 6
7 = Supelco TCL Mix 1
8 = From neat
9 = Supelco Single std
10 = Chem Service Single Standard
11 = Supelco single standard
12 = Supelco 8240B Mix 7
13 = Supelco single std
14 = Supelco single standard
15 = Neat Material
16 = Supelco Single standard
17 = Supelco Single standard
18 = Neat material
19= Supelco Single standard

At the time of this writing, these are the current standard solutions used for the calibration standards. However, these standards may change due to Vendor supply or project-specific requirements.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8277

Determination of Polynuclear Aromatic Hydrocarbon Compounds by Selective Ion Monitoring (SIM) Method 8270C and 8270D

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the determination of polynuclear aromatic hydrocarbon analytes in aqueous, soil, sediment, and other matrices by Selective Ion Monitoring (SIM) method. The SIM analysis scans for only a selected group of ions instead of the whole range of 35 to 500 m/z. The difference in scan time results in increased detector sensitivity, which allows us to report target analytes at very low levels.

1.1.2 This SOP follows SW-846 Method 8270D except for the specific deviations listed below or outlined in a project specific QAPP. This SOP is also applicable to SW-846 Method 8270C as all differences between the two revisions are clerical in nature. There are no technical differences.

1.1.3 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas/liquid/chromatography/mass spectroscopy and in the interpretation of chromatograms and mass spectra. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations and comments on Method 8270D, as published, which are followed as standard operating procedure in the performance of this method at Laucks:

1.2.1.1 Method 3611B (Alumina Column Cleanup and Separation of Petroleum Wastes) is utilized routinely for all soil/sediment sample extracts. Alumina cleanup is performed on aqueous sample extracts as deemed necessary.

1.2.1.2 The 5 initial calibration levels have been established from 0.2 µg/mL to 8.0 µg/mL in order to demonstrate linearity for all target analytes and to provide a low-level standard that will act as the reporting limit as outlined in the method.

1.2.1.3 Neutral surrogate compounds 1-Fluoronaphthalene, Fluorene-d10, and Pyrene-d10 are used routinely. Current surrogate recoveries are maintained in Laucks' quality control database.

1.2.1.4 The following compounds are used as spiking analytes:

2-Methylnaphthalene	Benzo(a)anthracene
Acenaphthylene	Chrysene
Acenaphthene	Benzo(b)fluoranthene
Fluorene	Benzo(k)fluoranthene
Phenanthrene	Benzo(a)pyrene
Anthracene	Indeno(1,2,3-cd)pyrene
Fluoranthene	Dibenz(a,h)anthracene
Pyrene	Benzo(g,h,i)perylene

1.2.1.5 Surrogates and matrix spikes are added to the sample such that the final amount injected from normally concentrated samples is 2.5 ng for all spiking analytes.

1.2.1.6 The concentration of internal standards added to all sample extracts and calibration standards has been decreased from 40 ng/µL per compound to 1.0 ng/µL as allowed by the method.

1.2.1.7 A 2 µL injection of all calibration standards, method blank, LCS, MS/MSD and sample extracts is performed. A 2 µL injection provides more mass on-column than a 1 µL injection, which improves the ability to confirm the presence of target analytes at low concentrations.

1.2.1.8 Laucks uses ±0.50 minutes relative to the daily CCV standard as an acceptable retention time window for internal standards in all analyses.

1.2.1.9 Laucks uses a relative response factor for all analytes. The method specifies that if the average RSD of all analytes for the initial calibration is ≤15%, then the RRF may be used for individual analytes with RSDs >15%. This method option will be used if any analyte's RSD exceeds 15% in the initial calibration. However, Laucks uses an internal guideline of 40 percent as an upper limit.

1.2.1.10 CLP Statement of Work, OLM04.2, DFTPP tuning criteria have been substituted as allowed by the method. Tuning criteria are listed in Appendix II.

1.2.1.11 All standards are stored at less than -10°C or by the manufacturer's recommendation. Sample extracts are stored at 4°C (±2°C).

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C (±2°C). Water samples must be extracted within 7 days and soil samples must be extracted within 14 days from the date of collection. All sample extracts must be analyzed within 40 days from the date of extraction.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on all NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard.
CCC	Calibration Check Compound. A compound of analytical interest whose RRF in the CCV is compared to the average RRF from the initial calibration. The % difference must be less than the value specified in the method for the CCV to be considered valid. CCCs must also meet maximum %RSD criteria in an initial calibration.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.

DFTPP	A solution containing DFTPP, pentachlorophenol, benzidine, and p,p'-DDT. An aliquot of this solution is analyzed at the start of a 12 hour QC period in order to verify DFTPP tuning criteria.
Internal Standard	A compound added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a know concentration, prior to analysis. Internal standards are used as the basis for quantitation of target compounds.
ICV	Initial continuing verification. A calibration standard obtained from a different manufacturer than the standards used for the initial and continuing calibrations that is analyzed after a compliant initial calibration.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts. A QC period is 12 hours starting with the injection of the DFTPP solution.
RF	Response Factor. The measure of the mass spectral response of an analyte compared to its internal standard. Response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RF is determined by the following equation:

$$RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$$

where:

A_x - Area of target analyte primary ion
C_{is} - Concentration of internal standard
A_{is} - Area of internal standard primary ion
C_x - Concentration of target analyte

RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the GC column.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.
SIM	Selective Ion Monitoring. This is a type of analysis when the MS detector is programmed to scan for only the selected ions.
Surrogate	Compounds added to every standard, blank, matrix spike, matrix spike duplicate, and sample at a known concentration; used to evaluate extraction and analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental samples.

2. Equipment List and Standards

2.1 Chromatographic System

Gas Chromatograph: Hewlett Packard 5890 I or II.

Carrier Gas: Helium 99.995% (high purity) or better.

Column: 30 m x 0.25 mm ID x 0.25 µm df capillary column (Restek RTX-5Sil MS or equivalent).

Automatic Sampler: Hewlett Packard 7673A with 19405A and 3396A controllers.

GC/MS Interface: Capillary direct to the ion source of the mass spectrometer, fixed temperature.

Mass Spectrometer: Hewlett Packard 5970B.

Data System: PC based Teknivent with ENVIROLINK data acquisition and processing software or HP Chemstation.

Miscellaneous: Assorted syringes, vials, caps, septa, injection port liners, ferrules, etc.

Note: All of the above equipment may be substituted with equivalent or better equipment.

2.2 Standards

2.2.1 All standards prepared from this operating procedure must be logged into one of two standards preparation logbooks. One is maintained for stock solutions prepared from neat chemicals or commercially prepared and certified standard solutions; the other is maintained for all working solutions. These logbooks are kept in the GC/MS semivolatile working area. When a standard is made, a solution number is assigned to it. This solution number is unique and will be used to track and identify the standard every time it is analyzed.

2.2.2 The following is an example of the solution nomenclature used for a working SIM PNA standard prepared on 11/13/91. The solution number assigned was MS2-77-2. This label represents the following:

MS	Solution was made and used as Mass Spec Standard
2	Solution was logged into standard book #2
77	Page number on which solution has been recorded
2	This denotes the second entry on page 77

2.2.3 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by purchasing solutions which have been fully documented by a commercial vendor.

2.2.4 Internal Standard Solution (SIM IS MIX @ 200 ug/mL).

Internal standards
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12
1,4-Dichlorobenzene-d4 (A)
(A) - this compound is present in the mix, but not appropriate to use for PNA analysis, and therefore, not reported.

2.2.4.1 Commercially prepared and certified internal standard solution is purchased at a concentration of 2000 µg/mL and is diluted 1:10. This standard is kept at -10°C until put into use. Once opened, the standard is kept at room temperature to avoid having the less soluble compounds fall out of solution. A new ampule of internal standard is opened on a weekly basis.

2.2.5 Preparation of DFTPP solution

2.2.5.1 Prepare a 25 µg/mL tuning solution which consists of DFTPP, pentachlorophenol, benzidine, and 4,4'DDT in methylene chloride. This solution is kept at -10°C.

2.2.6 Preparation of Calibration Standards

2.2.6.1 Calibration standards are prepared in methylene chloride from stock solutions that are purchased from a commercial source (e.g., Supelco or Restek). If an analyte required for calibration is not present in an available mixed solution, laboratory-prepared stock solutions that have been verified by GC/MS may be used. Laboratory prepared stock solutions should be tested against independent reference standards when they are available.

2.2.6.2 Calibration standards are prepared at five concentration levels. Standard concentrations at 0.2, 0.4, 1, 4 and 8 µg/mL are required. Each calibration standard contains all compounds of interest, surrogates, and internal standards.

2.2.7 Preparation of PNA stock solution at 200 µg/mL.

Stock Solution - PNA200
Total Volume 10ml

PNA Mix 2000 µg/ml	1ml	Supelco #47543-U
Carbazole 2000 µg/ml	1ml	Supelco #4-8076
PNA Surrogates 2000 µg/ml	1ml	Crescent Custom Mix #CCS-512
Methylene chloride	7ml	

2.2.7.1 Combine appropriate amounts of all solutions in a clean, 10 mL volumetric flask so that all analytes are present at 200 µg/mL. Dilute to volume with methylene chloride, stopper the flask and mix well. Record all information in the working standards logbook and transfer the contents of the volumetric flask into an amber screw-cap vial. Store this solution in the freezer when not in use. The vial must be marked with the logbook name, standard type, preparation date, solvent used, expiration date, and preparer initials.

2.2.7.2 The vendor and catalog numbers provided are for reference only. Other vendor's certified solutions may be substituted.

2.2.8 Preparation of working calibration standards

2.2.8.1 To prepare working calibration standards, add the amounts listed below in (µL) of PNA200, internal standard solution (SIM IS @ 200 µg/mL) and methylene chloride to clean vial inserts and use within one week.

SIM PNA Calibration Curve

	Amount of **PNA200**	Amount of Methylene Chloride
Working PNA 8	8µl	192µl
Working PNA 4	4µl	196µl
Working PNA 1	1µl	199µl

Intermediate PNA solution
1 µl **PNA200** in 199µl methylene chloride

	Amount of Intermediate Solution	Amount of Methylene Chloride
Working PNA 0.4	80µl	120µl
Working PNA 0.2	40µl	160µl

2.2.8.2 The working PNA 1 standard is the continuing calibration solution. The continuing calibration solution must be prepared on a weekly basis.

2.2.8.3 Calibration stock solutions which are received sealed in ampules from the manufacturer are useable up to their manufacturer's expiration date. The mixed PNA200 stock solution must be prepared once every twelve months, or sooner, if standards have degraded or concentrated such as evidenced by a significant increase in response for all analytes in the standard.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 All waste solvents, expired standards and old extracts should be disposed of in the solvent waste can located in the prep area fume hood. Refer to LTL-2001, *Waste Segregation and Disposal* for more detail.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Chromatographic Conditions

Initial Temperature:	80°C.
Initial Time:	1 minutes.
Column Temperature Program:	80°-250°C at 40 °C /minute, 250-320 at 20 °C /minute
Final Temperature:	320°C for 5.5 minutes.
Injector Temperature:	275-300°C.
Transfer Line Temperature:	280-310°C.
Injector Purge Time Off:	1 minute.
Injection Volume:	2 µL.
Column Linear Velocity:	30-40 cm/sec (nominal 35 cm/sec measured at 30°C).

4.1.2 Mass Spectrometer Conditions

Electron Energy:	70 volts.
Scan Time:	Not to exceed 1 second per scan.
Scan Start Time:	3.5 minutes.
Scan Time Range:	from 3.5 to 6.5 minutes.
Scans in Mass Segment:	125 to 177 amu.
Scan Time Range:	from 6.5 to 10.8 minutes.
Scans in Mass Segment:	165 to 245 amu.
Scan Time Range:	from 10.8 to 15 minutes.
Scans in Mass Segment:	248 to 280 amu.

4.2 Method Detection Limit Study

4.2.1 MDL studies will continue to be performed. This procedure is fully described in Laucks SOP on Determination of MDL's.

4.2.2 MDLs for this method are in the range of 0.005 – 0.1 µg/L for waters and 0.4 – 2.6 µg/kg for soils.

4.3 Method Reporting Limits

4.3.1 The reporting limits for this method are determined by the low level standard. Reporting limits are listed in Appendix VII.

4.4 Method Validation

4.4.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher. This procedure is fully described in Laucks SOP on Determination of Precision and Accuracy Studies.

4.5 Routine Instrument Maintenance

4.5.1 Instrument maintenance may be required for good chromatography and proper calibration. The following steps may be undertaken if needed before the analysis of any standard or sample:

A. Cool the GC oven to 30°C.
B. Check background for the instrument using manual tune to determine the operating status of the instrument.
C. Remove the injector septum and quartz liner.
D. Remove the column from the injector.
E. Install a new column ferrule.
F. Clip off 10 cm of the column. Check for a proper cut.
G. Reinstall the column and adjust the alignment.
H. Install a clean quartz liner.
I. Reinstall the O-ring or replace it if worn.
J. Install a new septum.
K. Clean and reinstall the autosampler syringe.

4.5.2 After any of these steps have been taken, recheck background, correct any leaks by tightening fitings, then ramp the GC oven twice from 50° to 310°C at 20° /minute for 10 minutes.

4.6 Instrument Tuning

4.6.1 The HP 5970B mass spectrometer uses FC-43 (PFTBA) as a mass calibration compound. Each instrument will require different ion ratios to pass the required DFTPP performance criteria. See Appendix II for the DFTPP tuning criteria. The following ratios are therefore approximate. Use manual tuning to tune the mass spectrometer.

Ion	% of ion 69
69	100%
131	25-35%
219	25-35%
414	1-3%

4.6.2 Ion peak widths should be in the 0.5 to 0.6 amu range. Excessive peak width can lead to loss of minor isotope peaks. Insufficient peak width can result in the loss of sensitivity. Ions for water, nitrogen, and oxygen should be <5% of ion 69.

4.6.3 Analyze an aliquot of the DFTPP tuning solution in order to verify DFTPP tuning criteria, degradation, and column tailing factors. The spectrum of the DFTPP must pass tuning criteria before any other standards are injected. Retuning of the instrument may be necessary to achieve this. Additionally, the DFTPP spectrum should be well within the performance criteria - i.e., no ion abundance should be borderline and the ratios should be routinely reproducible to insure a stable calibration.

4.6.4 The DFTPP tuning solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no visible peak tailing, as demonstrated by the peak tailing factor. The calculation of peak tailing factors is illustrated in Appendix XIII. The acceptance criteria for benzidine is <3.0 and pentachlorophenol is <5.0. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 inches of the capillary column.

4.6.5 Analysis of the DFTPP must meet the ion abundance criteria listed in Appendix II before initiation of initial or continuing calibration standard analysis.

4.7 Initial and Continuing Calibration Standards and Initial Calibration Verification

4.7.1 After demonstrating compliance with the DFTPP tuning criteria, analyze the calibration standards within 12 hours.

4.7.2 Check the integrations and retention times of all compounds in each standard. Examine the peaks to ensure that peak saturation and/or chromatographic overload has not occurred in the upper calibration standard.

4.7.3 All compounds in the initial and continuing calibration standards must meet the criteria listed in Appendix IV and Appendix V, respectively.

4.7.4 After completion of a compliant initial calibration curve, an initial calibration verification (ICV) may be analyzed. This standard is from a source different than that of the calibration standards. All analyte recoveries should be within 70-130% of the expected value with up to 2 analytes exceeding this control limit due to sporadic marginal failures.

4.8 Sample Analysis

4.8.1 After completion of a compliant continuing calibration standard, sample analysis may be performed.

4.8.2 Remove the sample extracts from the refrigerator and allow to warm to room temperature.

4.8.3 Transfer 200 µL of extract to a vial insert.

4.8.4 Add 2 µL of IS solution (SIM IS MIX @ 200 ug/mL).

4.8.5 Cap the vial and mix well.

4.8.6 Place the vial onto the autosampler for analysis.

4.8.7 For dilutions, decide what dilution is necessary from previous data or analyst judgment. Make dilution and then add 2 µL of SIM IS solution prior to capping vial. Mix well prior to analysis.

4.8.8 In general, the method blank accompanying the samples is injected prior to the analysis of the samples. This is not a requirement, however. Samples are then injected until the end of the 12 hour clock.

4.8.9 Compound Identification

4.8.10 Compounds are tentatively identified if a peak elutes within 0.5 minute of that compound in the CCV standard. In addition, the internal standard for that compound should also be within 0.5 minute of its counterpart in the CCV standard. To confirm the presence of that compound in the sample extract, the mass spectrum of the peak must be evaluated. Spectra are compared against standard spectra of each compound generated on the instrument used for analysis.

4.8.10.1 The following criteria are used to evaluate mass spectra.

4.8.10.1.1 The intensities of the characteristic ions of a compound maximize within one scan of each other. Searches performed based on the presence of a target compound at a compound-specific retention time will be accepted as meeting this criterion.

4.8.10.1.2 All ions present in the standard mass spectra at a relative intensity greater than 30% (most abundant ion in the spectrum equals 100%) or the three ions of greatest intensity must be present in the sample spectrum.

4.8.10.1.3 The relative intensities of ions specified must agree within 30% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.

4.8.10.1.4 If a compound cannot be verified by all of these criteria, but in the technical judgment of the analyst, the identification is correct, then report that identification and proceed with quantification.

4.8.10.1.5 The experienced analyst's judgment weighs heavily in evaluating chromatograms and mass spectra for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. If this has occurred, it must be fully documented in the appropriate report notes.

4.8.11 Common Analytical Problems

4.8.11.1 An analyst's professional judgment plays a large role in how data is interpreted. The following guidelines should be followed in order to facilitate consistency between analysts. Any anomalies not addressed in this SOP must be discussed with the supervisor prior to implementation. All anomalies and corrective actions must be documented.

4.8.12 Carryover

4.8.12.1 In some cases, if analytes in a sample are very high, there may be carryover from one analysis to the next. If a sample is suspected of being high due to historical data or extract color, the sample should be diluted prior to analysis, or one or more blanks should be analyzed after the sample to insure that there is no carryover between analyses.

4.8.12.2 However, in the case where high levels were not expected, and do appear in a sample analysis, the analysis after it should be examined carefully for carryover.

4.9 Manual Peak Integrations

4.9.1 Manual peak integrations should be used only when necessary to correct for matrix interference, tangent peaks, and rising baselines. Manual integrations are not to be used in an attempt to meet calibration, surrogate recovery, or spike recovery criteria. Refer to LTL-1020, *The Integration of IC, GC, HPLC, and GC/MS Peaks* for more detail.

4.9.2 If the chromatogram shows degradation due to sample loading e.g. split peaks, lift off, or severe tailing, the sample should be diluted and reanalyzed if required detection limits permit.

4.9.3 If a manual integration is necessary, use the following guidelines:

- Integrate only the peak. Start where the peak lifts from the baseline and end as soon as it returns to the baseline. Do not integrate extra baseline in an attempt to increase peak area.
- Do not "peak shave". Do not cut off legitimate parts of the peak in order to reduce peak area.
- In cases of tangent skims, do not increase or decrease peak areas or heights by skimming extra long baselines or drawing the baseline too low.
- Always initial and date your manual integrations.

4.10 Compound Quantification

4.10.1 Target compound concentrations are calculated using the following equations:

$$\text{Sample Concentration (ng/}\mu\text{L)} = \frac{A_x \times C_{is}}{A_{is} \times RF_{average}}$$

where:

A_x = Response for the target analyte
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard, in ng/µL
$RF_{average}$ = Average Response Factor (calculated from the initial calibration)

4.10.2 If the %RSD of a compound's relative response factor is 15% or less, than the concentration may be determined using the average response factor from the initial calibration. Alternatively, the RF may be used as long as the average RSD of all calibration analytes is ≤15%. Laucks uses 40% percent as the upper limit for all compounds as an internal guideline although it is not a requirement of SW 846.

4.10.3 The above equation is used directly by the HP computer to yield the extract concentration.

4.10.4 To calculate the actual sample concentration, the following calculation must be used.

Aqueous samples:

$$\text{Extract Concentration (}\mu\text{g/L)} = \frac{F \times D \times V_f \times GPC}{V_i}$$

where:

F = Amount found from HP quantitation report (ng/µL)
D = Dilution factor of extract
V_f = Final extract volume (µL)
GPC = GPC dilution factor. Use 1 if GPC was not performed, 2 if GPC was used
V_i = Initial sample volume (mL)

Non-aqueous samples:

$$\text{Sample Concentration}(\mu g/kg) = \frac{F \times D \times V_f \times GPC}{W_s \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

4.10.5 Normally, these calculations are automatically performed by the LIM system.

4.10.6 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

5. Quality Control

5.1 General Issues

5.1.1 See Appendix VIII and IX for QA/QC limit and corrective action tables.

5.2 Initial Calibration

5.2.1 Criteria

5.2.1.1 Initial calibration data are evaluated using %RSD of the relative response factors as well as minimum average RRFs. The %RSD must be ≤ 30% for each individual Calibration Check Compound (CCC). All other RRFs must be ≤ 15%. Alternatively, RRFs may be used as long as the average RRFs for all compounds in the method are ≤ 15%. Laucks uses an internal guideline of 40% as an upper control limit for each compound.

5.2.1.2 Calculate the %RSD for all compounds. CCC criteria are listed in Appendix V.

5.2.1.3 Calculate the individual and average RFs for each compound.

5.2.1.4 RFs are calculated using the equation

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Response of target analyte
A_{is} = Response of internal standard
C_s = Amount of target analyte (ng/µL)
C_{is} = Amount of internal standard (ng/µL)

5.2.1.5 The minimum acceptable average RF for all analytes is 0.010.

5.3 Continuing Calibration Verification

5.3.1 Criteria

5.3.1.1 At the beginning of each 12 hour QC period, a CCV standard is analyzed. The RF for each compound is calculated and the percent difference is calculated as follows:

$$\%Difference = \frac{RF_c - RF_i}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration.
RF_c = RRF from CCV standard.

5.3.1.2 The %D results for all CCCs must be ≤20%. There are no guidelines for the %D for all other compounds, however Laucks uses 40% as an internal guideline.

5.3.1.3 In addition, all compounds must have a minimum RRF of at least 0.010. See Appendix V for all CCV criteria.

5.3.2 Corrective action

5.3.2.1 Check that all peaks have been properly integrated. Check the calculations. If the %D criteria are still not met, perform corrective instrument maintenance or re-tuning, and reanalyze the standard. If the %D or minimum RF criteria are still not met, a new CCV standard may need to be prepared, a new initial calibration performed, a new column installed, or other instrument maintenance performed in order to meet the CCV criteria.

5.4 Method Blank

5.4.1 Criteria

5.4.1.1 A method blank is used to verify contamination-free reagents and apparatus. A method blank is prepared with every set of samples extracted at the same time, at a frequency of at least one blank per 20 samples.

5.4.1.2 Laucks criteria for a method blank is less than one-half the RL for each analyte, or less than 10 percent of the regulatory limit associated with that analyte, or less than 10 percent of the sample result for the same analyte, whichever is greater for the method blank to be acceptable. Method blank control limits are detailed in Appendix VI.

5.4.2 Corrective action

5.4.2.1 If an analyte is found in the blank but not in any of the associated samples, the sample batch may not require re-extraction unless this is required by contractual obligations. Consult the QC officer to determine if re-extraction and reanalysis are required.

5.4.2.2 If any analyte exceeds the control limit in the blank and is also found in the associated sample extracts, the samples must be re-extracted and reanalyzed.

5.4.2.3 In any event, it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

5.5 QC Check Sample (LCS)

5.5.1 Criteria

5.5.1.1 The LCS is used to determine whether a method is in control during sample preparation and analysis. A LCS follows the same protocol as the matrix spike analysis except that the spiking solution is added to a blank matrix (deionized water for water or sea sand for soil/sediments) instead of an actual sample. A Lab Control Sample is prepared with every set of samples extracted at the same time, at a frequency of at least one LCS per 20 samples. At the time of the writing of this SOP, the established control limits are 20-160% for all spiking compounds.

5.5.2 Corrective action

5.5.2.1 Check all peak integrations and sample calculations.

5.5.2.2 If the analyte recoveries still exceed the control limits, the blank spike extract may be reanalyzed.

5.5.2.3 If the analyte recoveries are still out of control, re-extraction of the associated samples is required.

5.5.3 Matrix Spike

5.5.3.1 Criteria

5.5.3.2 A sample is chosen from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is not required that a matrix spike analysis be performed with each extraction batch unless the project QAPP requires it. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of selected target analytes. Analyte recovery is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

5.5.3.3 At the time of the writing of this SOP, the established control limits are 20-160% for all spiking compounds.

5.5.4 Corrective action

5.5.4.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases, a narrative explanation of the condition is required to detail the corrective actions taken.

5.6 Matrix Spike Duplicate

5.6.1 Criteria

5.6.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values.

5.6.1.2 This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2)/2} \times 100$$

where:

S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

5.6.1.3 At the time of the writing of this SOP, the established control limits for MS/MSD recoveries are 20-160% and 30 for the RPD values.

5.7 Surrogate Recovery

5.7.1 Criteria

5.7.1.1 Surrogates are chemically similar compounds added to every sample, method blank and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follow:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:

S_m = Concentration of surrogate measured in extract.
S_a = Concentration of surrogate added.

5.7.1.2 At the time of the writng of this SOP, the established control limits for surrogate recoveries are 20-160%.

5.7.2 Corrective Action

5.7.2.1 Check calculations for possible error.

5.7.2.2 Check instrument performance, if necessary correct the problem and re-analyze the extract.

5.7.2.3 Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant matrix interference. This may result in the dilution of the surrogate response to the point that the recoveries can not be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. For all data package work both the diluted and undiluted analyses will be provided.

5.7.2.4 Re-extraction is not necessary in the case where a sample is chosen for MS/MSD analyses and the same recovery pattern is present in all three analyses. In this case, matrix effects may be assumed and re-extraction is not required for this sample.

5.7.2.5 All other circumstances require re-extraction and reanalysis of the affected sample(s).

5.7.2.6 Out of control surrogate recoveries in the method blank require that all the samples in the associated batch be re-extracted and reanalyzed. It is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

6. Reports

6.1 Data Packet Organization

6.1.1 See Appendix I for a check list detailing data packet organization.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U: The analyte of interest was not detected, to the limit of detection indicated.

B: The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.

J: The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.

D: The value reported is derived from the analysis of a diluted sample or sample extract.

X: Indicates an unresolvable isomeric pair. This flag indicates that calculated results are the sum of the two isomers.

E: The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Edition, Method 8270D, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique," Revision 4, January 1998, U.S. EPA.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Edition, 2nd Update, Method 3611B, "Alumina Cleanup," Revision 4, January 1998, US EPA.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Edition, 2nd Update, "Method 8000B, Determinative Chromatographic Separations", Revision 2, December 1996, U.S.EPA.

US EPA. Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, Document Number OLM04.2, May 1999.

APPENDIX I

Data Packet Check List

QC Summary
Surrogate Recovery Summary Report
Blank Spike Recovery Report
MS/MSD Recovery Report
Method Blank Summary
GC/MS Instrument Performance Check
Internal Standard Area and RT Summary Report

Sample Data
Target Compound Results (Organics Analysis Data Sheet)
Sample Chromatograms, quantitation reports and spectra for all samples

Standards Data
Initial Calibration Summary Report
Chromatograms and quantitation reports for all initial calibration standards
Continuing Calibration Summary Reports
Chromatograms and quantitation reports for all CCV Standards

Raw QC Data
Bar Graph Spectrum, mass listing and chromatogram for every DFTPP injection
Method Blank Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all method blanks
Blank Spike Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all blank spikes
Matrix Spike/Matrix Spike Duplicate Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all MS analyses

Bench Sheets
All extraction bench sheets, analysis log book pages, chains of custody, and any other pertinent information.

APPENDIX II

DFTPP Tuning Criteria

Mass	Relative Abundance
51	30-80% of mass 198
68	less than 2% of 69
69	present
70	less than 2% of 69
127	25-75% of mass 198
197	less than 1% of 198
198	100%
199	5-9% of mass 198
275	10-30% of mass 198
365	greater than 0.75% of mass 198
441	0-100 % of mass 443
442	40-110% of mass 198
443	15-24% of mass 442

CLP requires that the three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak.

SW 846 states that the following approach has been shown to be useful: the mass spectrum of DFTPP may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other documented approaches suggested by the instrument manufacturer.

APPENDIX III

Calibration Standard Solution Concentrations, ng/µL

Compound	STD1	STD2	STD3	STD4	STD5
1-Fluoronaphthalene	0.2	0.4	1.0	4.0	8.0
Naphthalene	0.2	0.4	1.0	4.0	8.0
2-Methylnaphthalene	0.2	0.4	1.0	4.0	8.0
Acenaphthylene	0.2	0.4	1.0	4.0	8.0
Acenaphthene	0.2	0.4	1.0	4.0	8.0
Fluorene-d10	0.2	0.4	1.0	4.0	8.0
Fluorene	0.2	0.4	1.0	4.0	8.0
Phenanthrene	0.2	0.4	1.0	4.0	8.0
Anthracene	0.2	0.4	1.0	4.0	8.0
Carbazole	0.2	0.4	1.0	4.0	8.0
Fluoranthene	0.2	0.4	1.0	4.0	8.0
Pyrene-d10	0.2	0.4	1.0	4.0	8.0
Pyrene	0.2	0.4	1.0	4.0	8.0
Benzo(a)anthracene	0.2	0.4	1.0	4.0	8.0
Chrysene	0.2	0.4	1.0	4.0	8.0
Benzo(b)fluoranthene	0.2	0.4	1.0	4.0	8.0
Benzo(k)fluoranthene	0.2	0.4	1.0	4.0	8.0
Benzo(a)pyrene	0.2	0.4	1.0	4.0	8.0
Indeno(1,2,3-cd)pyrene	0.2	0.4	1.0	4.0	8.0
Dibenz(a,h)anthracene	0.2	0.4	1.0	4.0	8.0
Benzo(g,h,i)perylene	0.2	0.4	1.0	4.0	8.0

APPENDIX IV

Initial Calibration Criteria

Calibration Check Compound	%RSD Limit
Acenaphthene	≤30%
Fluoranthene	≤30%
Benzo(a)pyrene	≤30%

$RRF_{average}$ for all compounds must be < 15%.

APPENDIX V

Continuing Calibration Verification Criteria

Calibration Check Compound	%D Limit
Acenaphthene	≤20%
Fluoranthene	≤20%
Benzo(a)pyrene	≤20%

%D for all other analytes should be within 40%.

APPENDIX VI

Method Blank Control Limits

Compound	Control Limits Water	Control Limits Soil
All compounds	<1/2RL*	<1/2RL*

* See Appendix VII

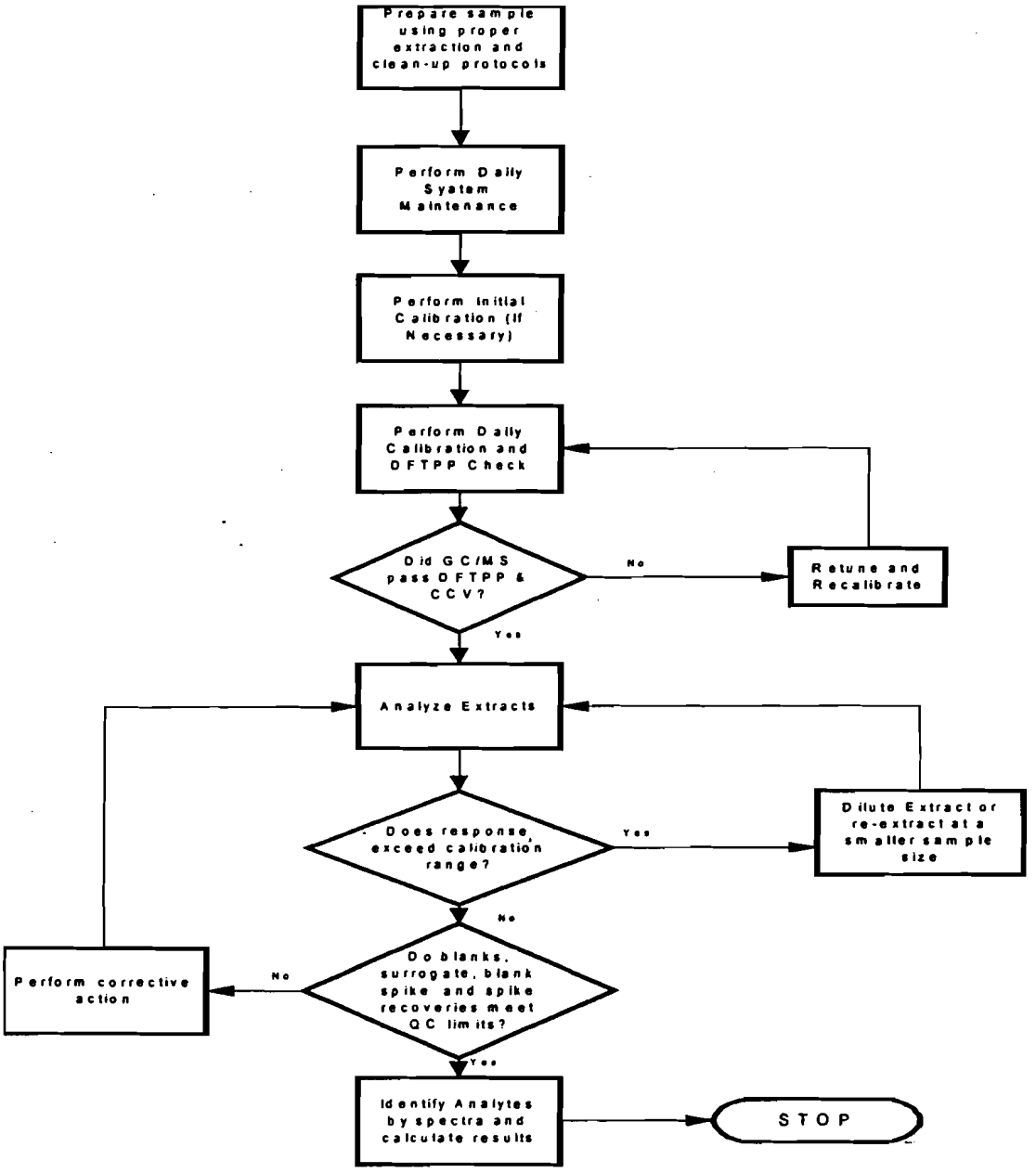
APPENDIX VII

Reporting Limits

Compound	Water µg/L	Soil µg/kg
2-Methylnaphthalene	0.1	7
Acenaphthylene	0.1	7
Acenaphthene	0.1	7
Fluorene	0.1	7
Phenanthrene	0.1	7
Anthracene	0.1	7
Carbazole	0.1	7
Fluoranthene	0.1	7
Pyrene	0.1	7
Benzo(a)anthracene	0.1	7
Chrysene	0.1	7
Benzo(b)fluoranthene	0.1	7
Benzo(k)fluoranthene	0.1	7
Benzo(a)pyrene	0.1	7
Indeno(1,2,3-cd)pyrene	0.1	7
Dibenz(a,h)anthracene	0.1	7
Benzo(g,h,i)perylene	0.1	7

APPENDIX VIII

Method 8270D Flow Chart



APPENDIX IX

Method 8270D QC Requirements and Corrective Actions

QC Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Minimum of 5 levels, lowest near but above MDL, %RSD for CCC ≤30, All others should be ≤15%.	5 levels, %RSD for CCC ≤30%, all others ≤15%, All RFs >0.01 Alternatively, average RSD must be <15%.	As necessary due to major instrument maintenance or continuing difficulties meeting the CCV requirements.	Re-analysis of out of control standards.	Copies of all raw data, mass calibration, tune, and Form VI.
Internal Standards	Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 40 ng/uL ea. Area must be -50% to +100% of the IS in the CCV. RT must be within ±0.06 RT units of the RT of the IS in the CCV.	Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 2.0 ng/ul ea. Area must be -50% to + 100% of the area of the IS in the CCV. RT must be within 0.5 minutes of RT in CCV.	All standards, blanks, matrix spikes, matrix spike duplicates, blank spikes, SRMs, and sample re-extracts.	Re-analyze any sample which IS areas or RTs are out of range.	Notation in case narrative if applicable.
DFTPP Tuning Verification (50 ng)	Mass Abundance 51 30-60% of 198 68 <2% of 69 69 present 70 <2% of 69 127 40-60% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >.1% of 198 441 present but < 443 442 >40% of 198 443 17-23% of 442	Mass Abundance 51 30-80% of 198 68 <2% of 69 69 present 70 <2% of 69 127 25-75% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >.75% of 198 441 present but < 443 442 40-110% of 198 443 15-24% of 442	Every 12 hours; CLP Criteria is used as allowed by the method.	Retune instrument to pass criteria.	Copy of DFTPP check with the file.
Continuing Calibration Verification	Mid-level standard every 12 hours. %D ≤20% for CCCs, all others ≤30.	1.0 ng/ul standard every 12 hours. %D for CCC≤20, all others ≤40.	Every 12 hours.	Perform system maintenance, re-analyze CCV standard.	Copies of raw data, mass calibration, tune, and Form VII.
Method Blank	Analytes must be <MDL	All analytes <1/2RL.	One method blank per 20 samples or each extraction batch of samples, whichever is more frequent.	Re-analyze blank. If still out of control, re-extract the entire batch of samples.	Notation in case narrative if applicable.
Surrogate Recovery	Upper and lower recovery limits determined by 3X standard deviation of at least twenty samples. To be performed at least semi-annually.	1-Fluoronaphthalene, Fluorene-d10, Pyrene-d10 surrogates are used. Limits are updated annually.	All samples, method blanks, blank spike, MS/MSD.	Re-extraction of the sample is required if any surrogate is out. All surrogates must be in control in the method blank otherwise all associated samples must be re-extracted..	Any out of control surrogates are to be documented in the job comments section or case narrative.

QC Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Blank Spike Recovery	Empirically derived from 20 or more BS. QC interval equals 3X standard deviation.	Empirically derived from 20 BS. QC interval equals 3X standard deviation.	One per batch of twenty.	Re-extract batch unless MS/MSD is perfect.	Narrate in case narrative.
MS/MSD Recovery & RPD	Empirically derived from 10 or more MS/MSD pairs. QC interval equals 3X standard deviation.	Empirically derived from 10 or more MS/MSD pairs. QC interval equals 3X standard deviation. Control limits: 20-160%	One per batch of twenty.	Re-extract batch unless blanks spike is perfect or documentable matrix effect is present.	Narrate problems in case narrative.

APPENDIX X

Analytes Ammenable to Analysis by Method 8270D SIM

Current 8270 SIM PNA Target Analytes:

Compound	Primary Ion	Secondary Ion(s)
Naphthalene	128	129,127
2-Methylnaphthalene	142	141
Acenaphthylene	152	151,153
Acenaphthene	153	154,152
Fluorene	166	165,167
Phenanthrene	178	179,176
Anthracene	178	176,179
Carbazole	167	166
Fluoranthene	202	200
Pyrene	202	200,203
Benzo(a)anthracene	228	229,226
Chrysene	228	226,229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Indeno(1,2,3-cd)pyrene	276	274
Dibenzo(a,h)anthracene	278	279
Benzo(g,h,i)perylene	276	274,277
1-Fluoronaphthalene	146	147,145
Fluorene-d10	176	174,177
Pyrene-d10	212	

APPENDIX XI

Elution Order of the Compounds

Compound	Retention Time (min)
Naphthalene	4.38
2-Methylnaphthalene	4.88
Acenaphthylene	5.56
Acenaphthene	5.69
Fluorene	6.08
Phenanthrene	6.90
Anthracene	6.94
Carbazole	7.08
Fluoranthene	8.07
Pyrene	8.34
Benzo(a)anthracene	9.77
Chrysene	9.83
Benzo(b)fluoranthene	11.27
Benzo(k)fluoranthene	11.30
Benzo(a)pyrene	11.78
Indeno(1,2,3-cd)pyrene	13.78
Dibenzo(a,h)anthracene	13.79
Benzo(g,h,i)perylene	14.36
1-Fluoronaphthalene	4.36
Fluorene-d10	6.06
Pyrene-d10	8.31

APPENDIX XII

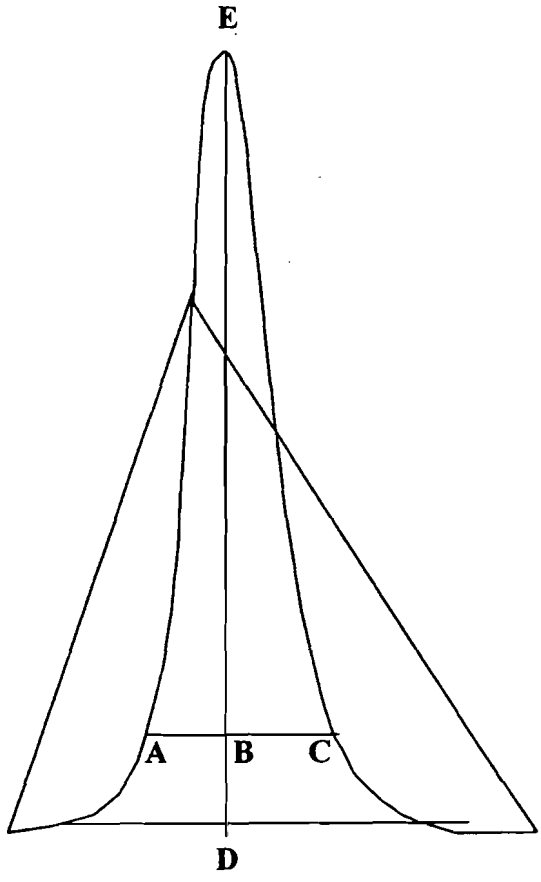
Semivolatile Internal Standards With Corresponding Analytes Assigned For Quantitation

Naphthalene-d8	**Acenaphthene-d10**
Naphthalene	Acenaphthylene
2-Methylnaphthalene	Acenaphthene
1-Fluoronaphthalene	Fluorene
	Fluorene-d10

Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
Phenanthrene	Pyrene	Benzo(b)fluoranthene
Anthracene	Benzo(a)anthracene	Benzo(k)fluoranthene
Carbazole	Chrysene	Benzo(a)pyrene
Fluoranthene	Pyrene-d10	Indeno(1,2,3-cd)pyrene
		Dibenzo(a,h)anthracene
		Benzo(g,h,i)perylene

Appendix XIII

Calculation of Peak Tailing Factors



Peak Tailing Factor = BC/AB

Sample calculation: Peak Height = DE =100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm
AB = 11mm
BC = 12mm
Tailing Factor = 12/11 = 1.1

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8279

Determination of Semivolatile Organic Compounds by SW-846 Methods 8270C and 8270D

Revision history:

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the determination of semivolatile organic analytes in aqueous, soil, sediment, and other matrices. This SOP follows SW-846 Method 8270D except for the specific deviations listed below or outlined in a project's specific QAPP. This SOP is also applicable to SW-846 Method 8270C as all differences between the two revisions are clerical in nature. There are no technical differences.

1.1.2 This method is restricted to use by, or under the supervision of, analysts experienced in the use of gas/liquid/chromatography/mass spectroscopy and in the interpretation of chromatograms and mass spectra. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations and comments of Method 8270D, as published, which are followed as standard operating procedure in the performance of this method at Laucks:

1.2.1.1 Method 3640A (Gel Permeation Chromatography cleanup) is utilized routinely for all soil/sediment sample extracts. GPC cleanup is performed on aqueous sample extracts as deemed necessary.

1.2.1.2 The initial calibration has been increased from five to six concentration levels in order to demonstrate linearity for all target analytes and to provide a low-level standard that will act as the reporting limit as outlined in the method.

1.2.1.3 The concentration of internal standards added to all sample extracts and calibration standards has been decreased from 40 ng/µL per compound to 20 ng/µL in order to accommodate 2 µL injection volumes as allowed by the method. This adjustment keeps the total nanograms injected the same and prevents overloading the analytical column.

1.2.1.4 Lauck's uses a relative response factor for all analytes. The method specifies that if the average RSD of all analytes for the initial calibration is ≤15%, then the RRF may be used for individual analytes with RSDs >15%. This method option will be used if any analyte's RSD exceeds 15% in the initial calibration. However, Laucks uses an internal guideline of 40 percent as an upper limit.

1.2.1.5 For Appendix IX and TCLP analyses, the compound 3-methylphenol (m-Cresol) coelutes with 4-methylphenol (p-Cresol). Since the spectrum and retention time for these two compounds are the same, these two compounds will be reported together as

the sum of these two compounds and with the assumption that 3-methylphenol has the same response factor as the 4-methylphenol present in the standards.

1.2.1.6 CLP Statement of Work, OLM04.1, DFTPP tuning criteria has been substituted as allowed by the method. Typical tuning criteria are listed in Appendix II.

1.2.1.7 All standards are stored at less than -10°C or by the manufacturer's recommendation. Sample extracts are stored at 4°C ±2°C.

1.2.1.8 The surrogate and matrix spikes will be added to the sample such that the final amount injected from normally concentrated samples is 100 ng for the base/neutral compounds and 150 ng for the acids. Acids will not be injected at 200 ng per injection to prevent exceeding the highest calibration standard of 160 ng.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ± 2°C. Water samples must be extracted within 7 days of collection. Soil samples must be extracted within 14 days of collection. All sample extracts must be analyzed within 40 days of sample preparation.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time sample extracts are prepared. Blank spikes are required on NFESC work. In the context of this SOP, a blank spike is the same as a QC check standard.
CCC	Calibration Check Compound. A compound of analytical interest whose RRF in the CCV is compared to the average RRF from the initial calibration. The % difference must be less than the value specified in the method for the CCV to be considered valid. CCCs must also meet maximum %RSD criteria in an initial calibration.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
ICV	Initial continuing verification. A calibration standard obtained from a different manufacturer than the standards used for the initial and continuing calibrations that is analyzed after a compliant initial calibration.
CLP	Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
DFTPP	A solution containing DFTPP, pentachlorophenol, benzidine, and p,p'-DDT. An aliquot of this solution is analyzed at the start of a 12 hour QC period in order to verify DFTPP tuning criteria.
Internal Standard	A compound added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a know concentration, prior to analysis. Internal standards are used as the basis for quantitation of target compounds.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
MDL Standard	Method detection limit standard. A standard prepared so that the concentrations of the target analytes are approximately 4x the empirically determined MDLs on an extract basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts. A QC period is 12 hours starting with the injection of the CCV standard or DFTPP performance evaluation.
RF	Response Factor. The measure of the mass spectral response of an analyte compared to its internal standard. Response factors are determined by analysis of standards and are used in the calculation of concentrations of analytes in samples. RF is determined by the following equation: $RF = \frac{A_x \times C_{is}}{A_{is} \times C_x}$ where: A_x - Area of target analyte primary ion; C_{is} - Concentration of internal standard; A_{is} - Area of internal standard primary ion; C_x - Concentration of target analyte
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the GC column.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

SPCC	System Performance Check Compound. A compound of analytical interest whose relative response factor (RRF) is monitored in the CCV for signs of chromatographic degradation. SPCCs generally have low RRFs and tend to decrease in response as the chromatographic system begins to deteriorate. They must meet a minimum RRF for initial and continuing calibrations.
EICP	Extracted ion current profile-a plot of ion abundance versus time (or scan number) for ion(s) of specified mass(es).
Surrogate	Compounds added to every standard, blank, matrix spike, matrix spike duplicate, and sample extract at a known concentration; used to evaluate extraction and analytical efficiency by measuring recovery. Surrogates are brominated, fluorinated, or isotopically labeled compounds not expected to be detected in environmental samples.

2. Equipment List and Standards

2.1 Chromatographic System

Gas Chromatograph:	Hewlett Packard 5890 I or II
Carrier Gas:	Helium 99.999% (ultra purity) or better
Column:	30 m x 0.25 mm x 0.25 μ capillary column (Restek RTX5SIL-MS or equivalent)
Automatic Sampler:	Hewlett Packard 7673A or 7673 with 19405A and 3396A controllers
GC/MS Interface:	Capillary direct to the ion source of the mass spectrometer, fixed temperature
Mass Spectrometer:	Hewlett Packard 5970B
Data System:	PC based Teknivent with ENVIROLINK data acquisition and processing software or HP Chemstation.
Miscellaneous:	Assorted syringes, vials, caps, septa, injection port liners, ferrules, etc.
Note:	All of the above equipment may be substituted with equivalent or better equipment

2.2 Standards

2.2.1 Preparation of Semivolatile Standards

2.2.1.1 All standards prepared from this operating procedure must be logged into one of two standards preparation logbooks. One is maintained for stock solutions prepared from

neat chemicals; the other is maintained for all working solutions. These logbooks are kept in the GC/MS semivolatile working area. When a standard is made, a solution number is assigned to it. This solution number is unique and will be used to track and identify the standard every time it is analyzed.

2.2.1.2 An example of the solution nomenclature used in a working ABN standard prepared on 11/13/91. The solution number assigned was MS 2-77-2. This label represents the following:

MS	Solution was made and used as a Mass Spec standard.
2	Solution was logged into standard book #2.
77	Page number on which solution has been recorded.
2	This denotes the second entry on page 77.

2.2.1.3 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by purchasing solutions which have been fully documented by a commercial vendor.

2.2.2 Preparation of Internal Standard Solution (ABN IS MIX @ 2000 ug/mL).

Internal Standards
1,4-Dichlorobenzene-d4
Naphthalene-d8
Acenaphthene-d10
Phenanthrene-d10
Chrysene-d12
Perylene-d12

2.2.2.1 Commercially prepared and certified internal standard solution is purchased at a concentration of 2000 µg/mL and is used undiluted. This standard is kept at -10°C until put into use. Once opened, the standard is kept at room temperature to avoid having the less soluble compounds fall out of solution. A new ampule of internal standard is opened on a weekly basis.

2.2.3 Preparation of DFTPP solution:

2.2.3.1 Prepare a 25 µg/mL tuning solution which consists of DFTPP, pentachlorophenol, benzidine, and 4,4'DDT in methylene chloride. This solution is kept at -10°C.

2.2.4 Prepartion of Calibration Standards

2.2.4.1 Calibration standards are prepared in methylene chloride from stock solutions which are purchased from a commercial source (e.g., Supelco or Crescent). If an analyte required for calibration is not present in an available mixed solution, laboratory-prepared stock

solutions which have been verified by GC/MS may be used. Laboratory prepared stock solutions should be tested against independent reference standards when they are available.

2.2.4.2 Calibration standards are prepared at six concentration levels (5, 10, 25, 40, 60 and 80 ng/µL). Each calibration standard contains all compounds of interest, surrogates, and internal standards. The internal standards are added so that they are present in all calibration standards at a concentration of 20 ng/µL each.

2.2.5 Preparation of ABN200 stock solution

2.2.5.1 Assemble the following solutions in order to prepare a combined stock solution which will contain all analytes of interest at a concentration of 200 µg/mL each:

Analyte Mix	Vendor	Stock #
Benzidines Mix 2	Crescent	CC2410
Base/Neutrals Mix 1	Crescent	CC2458
Acids Mix 3	Crescent	CC2471R
SV Mix w/ Pyridine& Carbazole	Crescent	CC2473
Acid Surrogate Mix	Supelco	4-7261
B/N Surrogate Mix	Supelco	4-7262

Note: 1,2-diphenylhydrazine is detected as azobenzene. Azobenzene may be substituted for 1,2-diphenylhydrazine as it is in many commercially purchased calibration mixes.

2.2.6 Preparation of APPEND200 stock solution

2.2.6.1 Assemble the following solutions in order to prepare a combined stock solution which will contain all analytes of interest at a concentration of 200 µg/mL each:

Analyte Mix	Vendor	Stock #
Appendix IX Semivolatiles (15)	Crescent	CC2289
Appendix IX Semivolatiles (9)	Crescent	CC2289A
Appendix IX Semivolatiles (10)	Crescent	CC2289B
Appendix IX 1,3,5-TNB	Crescent	CC2289C
Semivolatiles Mix 5	Crescent	8SM-008A
Pentachlorethane	Crescent	3347D.20

2.2.6.2 The vendor and catalog numbers provided are for reference only. Other vendor's certified solutions may be substituted.

2.2.6.3 Combine appropriate amounts of all solutions in a clean, 10 mL volumetric flask so that all analytes are present at 200 µg/mL. Dilute to volume with methylene chloride, stopper the flask and mix well. Record all information in the working standards logbook

and transfer the contents of the volumetric flask into an amber screw-cap vial. Store this solution in the freezer when not in use. The vial must be marked with the logbook name, standard type, preparation date, solvent used, expiration date, and preparer initials.

2.2.7 Preparation of working calibration standards

2.2.7.1 To prepare working calibration standards, add the amounts listed below (in µL) of ABN200 stock solution, internal standard solution (ABN IS MIX @ 2000 ug/mL) and methylene chloride to clean vial inserts and use within one week.

	Amount of Standard ABN200 in µl	Amount of Standard APPEND200 in µl	Amount of Internal Standard in µl	Amount of CH_2Cl_2 in µl
Working ABN 5	5	5	2	190
Working ABN 10	10	10	2	180
Working ABN 25	25	25	2	150
Working ABN 40	40	40	2	120
Working ABN 60	60	60	2	80
Working ABN 80	80	80	2	40

2.2.7.2 Log into the working solutions logbook all of the above information as the standards are made. Store all standards at less than -10°C for up to one week when not in use.

2.2.7.3 The ABN 25 standard is the continuing calibration standard and it will need to be prepared on a weekly basis.

2.2.7.4 Calibration stock solutions which are received sealed in ampules from the manufacturer are useable up to their manufacturer's expiration date. The mixed ABN200 and APPEND200 stock solution must be prepared once every twelve months, or sooner, if standards have degraded or concentrated.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 All waste solvents, expired standards and old extracts should be disposed of in the solvent waste can located in the prep area fume hood. Consult the laboratory SOP for more detail on waste disposal.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Chromatographic Conditions

Initial Temperature:	45°C
Initial Time:	4 minutes
Column Temperature Program:	8°C/minute
Final Temperature:	320°C
Final Time:	8 minutes
Injector Temperature:	250-300°C (instrument dependent)
Transfer Line Temperature:	280-310°C (instrument dependent)
Injector Purge Time Off:	0.15 minutes
Injection Volume:	2 µL
Column Linear Velocity:	30-40 cm/sec (nominal 35 cm/sec measured at 30°C)

4.1.2 Mass Spectrometer Conditions

Electron Energy:	70 volts
Mass Range:	35 to 500 amu
Scan Time:	Not to exceed 1 second per scan

4.2 Method Detection Limit Study

4.2.1 MDL studies are performed on an annual basis. This procedure is fully described in Laucks SOP on Determination of MDL's.

4.2.2 MDLs for this method are in the range of 0.27 – 2.5 µg/L for waters and 29.0 – 201 µg/kg for soils.

4.3 Method Reporting Limits

4.3.1 The method reporting limit for this method shall be set as described by method 8000B by using the lowest calibration standard as the method report limit. Values detected below this level will be reported but will be "J" flagged as outlined in Section 5.2 of this SOP.

4.3.2 Reporting limits for aqueous samples are 5 µg/L with the exception of benzoic acid and 2,4-dinitrophenol which are 10 µg/L. Reporting limits for non-aqueous samples are 330 µg/kg with the exception of benzoic acid and 2,4-dinitrophenol which are 670 µg/kg.

4.3.3 For the additional AppendIX analytes, the reporting limit for aqueous samples is 5 µg/L with the exception of a,a-dimethylphenylethylamine and famphur where the reporting limit is 10 µg/L. Reporting limits for non-aqueous samples are 330 µg/kg and 670 µg/kg respectively.

4.4 Method Validation

4.4.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher. This procedure is fully described in Laucks SOP on Determination of QC limits.

4.5 Daily Instrument Maintenance

4.5.1 Instrument maintenance may be required for good chromatography and proper calibration. The following steps may be undertaken if needed before the analysis of any standard or sample:

A. Cool the GC oven to 30°C.
B. Check the background for the instrument using manual tune to determine the operating status of the instrument.
C. Remove the injector septum and quartz liner.
D. Remove the column from the injector.
E. Install a new column ferrule.
F. Clip off 10 cm of the column. Check for a proper cut.
G. Reinstall the column and adjust the alignment.
H. Install a clean quartz liner.
I. Reinstall the O-ring or replace it if worn.
J. Install a new septum.
K. Clean and reinstall the autosampler syringe.

4.5.2 After any of these steps have been taken, recheck background, correct any leaks by tightening fitings, then ramp the GC oven twice from 50° to 310°C at 20° /minute for 10 minutes

4.6 Instrument Tuning

4.6.1 The HP 5970B mass spectrometer uses FC-43 (PFTBA) as a mass calibration compound. Each instrument will require different ion ratios to pass the required DFTPP performance criteria. See Appendix II for the DFTPP tuning criteria. The following ratios are therefore approximate. Use manual tuning to tune the mass spectrometer.

Ion	% of ion 69
69	100%
131	25-35%
219	25-35%
414	1-3%

4.6.2 Ion peak widths should be in the 0.5 to 0.6 amu range. Excessive peak width can lead to loss of minor isotope peaks. Insufficient peak width can result in the loss of sensitivity. Ions for water, nitrogen, and oxygen should be <5% of ion 69. A copy of the PFTBA spectrum and tabular listing should be kept with the instrument historical file.

4.6.3 The DFTPP tuning solution should also be used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD should not exceed 20%. Benzidine and pentachlorophenol should be present at their normal responses, with no visible peak tailing, as demonstrated by the peak tailing factor. The calculation of peak tailing factors is illustrated in Appendix XII. The acceptance criteria for benzidine is <3.0 and pentachlorophenol is <5.0. If degradation is excessive and/or poor chromatography is noted, the injection port may require cleaning. It may also be necessary to break off the first 6-12 inches of the capillary column.

4.6.4 Analysis of the DFTPP must meet the ion abundance criteria listed in Appendix II before initiation of initial or continuing calibration standard analysis.

4.7 Initial and Continuing Calibration Standards and Initial Calibration Verification

4.7.1 After demonstrating compliance with the DFTPP tuning criteria, analyze the calibration standards within 12 hours.

4.7.2 Check the integrations and retention times of all compounds in each standard. Examine the peaks to ensure that peak saturation and/or chromatographic overload has not occurred in the upper calibration standard.

4.7.3 All compounds in the initial and continuing calibration standards must meet the criteria

listed in Appendix IV and Appendix V, respectively.

4.7.4 After completion of a compliant initial calibration curve, an initial calibration verification (ICV) may be analyzed. This standard is from a source different than that of the calibration standards. At the time of the writing of this SOP, Laucks uses a commercially prepared and certified standard solution purchased from Radian International (Catalog # ERS072). This ICV standard is analyzed at a concentration range of 32-64 ug/mL which is higher than the CCV analyzed on a routine basis. All analyte recoveries should be within 70-130% of the expected value with up to ten analytes exceeding this control limit due to sporadic marginal failures.

4.8 Sample Analysis

4.8.1 After completion of a compliant continuing calibration standard, sample analysis may be performed.

4.8.2 Remove the sample extracts from the refrigerator and allow to warm to room temperature.

4.8.3 Transfer 200 µL of extract to a vial insert.

4.8.4 Add 2 µL of IS solution (ABN IS MIX @ 2000 µg/mL).

4.8.5 Cap the vial and mix well.

4.8.6 Place the vial onto the autosampler for analysis.

4.8.7 For dilutions, decide what dilution is necessary from previous data or analyst judgment. Make dilution and then add 2 µL of ABN IS solution prior to capping vial. Mix well prior to analysis.

4.8.8 In general, the method blank accompanying the samples is injected prior to the analysis of the samples. This is not a requirement, however. Samples are then injected until the end of the 12 hour clock.

4.9 Compound Identification

4.9.1 Compounds are tentatively identified if a peak elutes within 0.5 minute of that compound in the CCV standard. In addition, the internal standard for that compound should also be within 0.5 minute of its counterpart in the CCV standard. To confirm the presence of that compound in the sample extract, the mass spectrum of the peak must be evaluated. Spectra are compared against standard spectra of each compound generated on the instrument used for analysis.

4.9.2 The following criteria are used to evaluate mass spectra.

4.9.2.1 The intensities of the characteristic ions of a compound maximize within one scan of each other. Searches performed based on the presence of a target compound at a compound-specific retention time will be accepted as meeting this criterion.

4.9.2.2 All ions present in the standard mass spectra at a relative intensity greater than 30% (most abundant ion in the spectrum equals 100%) or the three ions of greatest intensity must be present in the sample spectrum.

4.9.2.3 The relative intensities of ions specified must agree within 30% between the standard and sample spectra. Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample ion abundance must be between 20 and 80 percent.

4.9.2.4 If a compound cannot be verified by all of these criteria, but in the technical judgment of the analyst, the identification is correct, then report that identification and proceed with quantification.

4.9.2.5 The experienced analyst's judgment weighs heavily in evaluating chromatograms and mass spectra for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. If this has occurred, it must be fully documented in the appropriate report notes.

4.10 Common Analytical Problems

4.10.1 An analyst's professional judgment plays a large role in how data are interpreted. The following guidelines should be followed in order to facilitate consistency between analysts. Any anomalies not addressed in this SOP must be discussed with the supervisor prior to implementation. All anomalies and corrective actions must be documented.

4.10.2 Interferences

4.10.2.1 Problems have been associated with the following compounds analyzed by this method.

4.10.2.2 Dichlorobenzidine and 4-chloroaniline can be subject to oxidative losses during solvent concentration.

4.10.2.3 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reactions in acetone solution, and photochemical decomposition.

4.10.2.4 N-nitrosodiphenylamine decomposes in the gas chromatograph inlet forming diphenylamine and, consequently, may be detected as diphenylamine.

4.10.3 Carryover

4.10.3.1 In some cases, if analytes in a sample are very high, there may be carryover from one analysis to the next. If a sample is suspected of being high due to historical data or extract color, the sample should be diluted prior to analysis, or one or more blanks should be analyzed after the sample to insure that there is no carryover between analyses.

4.10.3.2 However, in the case where high levels were not expected, and do appear in a sample analysis, the analysis after it should be examined carefully for carryover. Any sample analyte whose concentration exceeds 250 ng/µL in the extract may be considered a candidate for carryover.

4.10.4 Manual Peak Integrations

4.10.4.1 Manual peak integrations should be used only when necessary to correct for matrix interference, tangent peaks, and rising baselines. Manual integrations are not to be used in an attempt to meet calibration, surrogate recovery, or spike recovery criteria. Refer to LTL-1020, *The Integration of IC, GC, HPLC, and GC/MS Peaks* for more detail.

4.10.4.2 If the chromatogram shows degradation due to sample loading e.g. split peaks, lift off, or severe tailing, the sample should be diluted and reanalyzed if required detection limits permit.

4.10.4.3 If a manual integration is necessary, use the following guidelines:

- Integrate only the peak. Start where the peak lifts from the baseline and end as soon as it returns to the baseline. Do not integrate extra baseline in an attempt to increase peak area.
- Do not "peak shave". Do not cut off legitimate parts of the peak in order to reduce peak area.
- In cases of tangent skims, do not increase or decrease peak areas or heights by skimming extra long baselines or drawing the baseline too low.
- Always initial and date your manual integrations.

4.11 Compound Quantification

4.11.1 Target compound concentrations are calculated using the following equations:

4.11.2 The equation for internal standard calculations is

$$Extract\ Concentration(ng / \mu L) = \frac{A_x \times C_{is}}{A_{is} \times RF_{average}}$$

where:

A_x = Response for the target analyte
A_{is} = Response for the internal standard
C_{is} = Concentration of internal standard, in ng/µL
$RF_{average}$ = Average Response Factor (calculated from the initial calibration)

4.11.3 If the %RSD of a compound's relative response factor is 15% or less, than the concentration may be determined using the average response factor from the initial calibration. Alternatively, the RF may be used as long as the average RSD of all calibration analytes is ≤15%. Laucks uses 40% percent as the upper limit for all compounds as an internal guideline although it is not a requirement of SW 846.

4.11.4 The above equation is used directly by the HP computer to yield the extract concentration.

4.11.5 Aqueous samples

4.11.5.1 To calculate the actual sample concentration, the following calculation must be used.

$$\text{Extract Concentration } (\mu g/L) = \frac{F \times D \times V_f \times GPC}{V_i}$$

where:

F = Amount found from HP quantitation report (ng/µL)
D = Dilution factor of extract
V_f = Final extract volume (µL)
GPC = GPC dilution factor. Use 1 if GPC was not performed, 2 if GPC was used
V_i = Initial sample volume (mL)

4.11.5.2 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

4.11.6 Non-aqueous samples

4.11.6.1 The results calculation for non-aqueous samples is very similar to that for aqueous

samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$\text{Sample Concentration}(\mu g/kg) = \frac{F \times D \times V_f \times GPC}{W_s \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

4.11.6.2 Any sample extracts which exceed the upper calibration range for any analyte of interest must be diluted and reanalyzed to bring the analyte into the working range of the calibration.

5. Reports

5.1 Data Packet Organization

5.1.1 See Appendix III for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Sample Result Reports

5.3.1 Data Qualifying Flags

5.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U: The analyte of interest was not detected, to the limit of detection indicated.

B: The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.

J: The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.

D: The value reported is derived from the analysis of a diluted sample or sample extract.

X: Indicates an unresolvable isomeric pair. This flag indicates that calculated results

are the sum of the two isomers.

E: The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.

6. Quality Control

6.1 General Issues

6.1.1 See Appendices VII and VIII for flow chart and QA/QC limit and corrective action tables.

6.2 Initial Calibration

6.2.1 Criteria

6.2.1.1 Initial calibration data are evaluated using %RSD of the relative response factors as well as minimum average RRFs. The %RSD must be ≤ 30% for each individual Calibration Check Compound (CCC). All other RRFs must be ≤15%. Alternatively, RRFs may be used as long as the average RRFs for all compounds in the method are ≤15% and the CCCs are ≤30%. Laucks uses an internal guideline of 40% as an upper control limit for each compound.

6.2.1.2 Calculate the %RSD for all compounds. CCC criteria are listed in Appendix IV.

6.2.1.3 Calculate the individual and average RFs for each compound.

6.2.1.4 RFs are calculated using the equation

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Response of target analyte
A_{is} = Response of internal standard
C_s = Amount of target analyte (ng/µL)
C_{is} = Amount of internal standard (ng/µL)

6.2.1.5 Laucks has established additional criterion by requiring a minimum acceptable average RRF for all non SPCC analytes of 0.010. At this time the Appendix IX compounds have no minimum RRF. See Appendix IV for a listing of all SPCCs.

6.2.2 Corrective action

6.2.2.1 If the criteria are not met, other standards may be analyzed or appropriate instrument maintenance and analysis of new standards must be performed. Failure to achieve the required minimum average RRF for the SPCC compounds may indicate the need to perform instrument maintenance or prepare fresh calibration standards.

6.2.2.2 Some compounds, including but not limited to the nitroanilines, the nitrophenols and some additional Appendix IX analytes, tend to show poor or erratic response at the lower calibration levels.

6.2.2.3 For routine analyte lists the lowest standard may be omitted from the initial calibration as long as the %RSD does not exceed 30% for any which are CCCs. Additionally, those compounds which are omitted from the lower calibration levels will also have an elevated reporting level equivalent to that of the lowest standard.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 At the beginning of each 12 hour QC period, a CCV standard at a concentration level of 25 µg/mL is analyzed. The RF for each compound is calculated and the percent difference is calculated as follows:

$$\%Difference = \frac{RF_c - RF_i}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration.
RF_c = RRF from CCV standard.

6.3.2 The %D results for all CCCs must be less than or equal to 20%. Laucks uses 40% percent as the upper limit for all compounds as an internal guideline although it is not a requirement of SW 846.

6.3.2.1 In addition, all compounds (with the exception of the Appendix IX analytes) must have a minimum RRF of at least 0.010 compounds. See Appendix V for all CCV criteria.

6.3.3 Corrective action

6.3.3.1 Check that all peaks have been properly integrated. Check the calculations. If the %D criteria are still not met, perform corrective instrument maintenance or re-tuning, and reanalyze the standard. If the %D or minimum RF criteria are still not met, a new CCV

standard may need to be prepared, a new initial calibration performed, a new column installed, or other instrument maintenance performed in order to meet the CCV criteria.

6.4 Method Blank

6.4.1 Criteria

6.4.1.1 A method blank is used to verify contamination-free reagents and apparatus. A method blank is prepared with every set of samples extracted at the same time, at a frequency of at least one blank per 20 samples.

6.4.1.2 Laucks criteria for a method blank is less than one-half the RL for each analyte or less than 10 percent of the regulatory limit associated with that analyte, or less than 10 percent of the sample result for the same analyte.

6.4.1.3 Method blank corrective action limits are detailed in Appendix VI.

6.4.2 Corrective action

6.4.2.1 When these criteria are exceeded, corrective action should be taken to find/reduce/eliminate the source of this contamination in the method blank. Sample corrective action may be limited to qualification for blank contamination. When the concentrations of any target analytes within the method blank are above one-half of the reporting limit, the effect this may have had on the samples will be assessed.

1) If any analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps shall be taken to find/reduce/eliminate the source of this contamination in the method blank. The case narrative should also discuss the situation.

2) Re-analysis of the method blank and any samples containing the same contaminant(s) which exceeded the corrective action limit(s) is performed if an analyte is found in the method blank and some, or all, of the other batch samples. If the contamination remains, the contaminated samples of the batch would be reprepared and reanalyzed with a new method blank and batch specific QC samples.

3) Sporadic cases of contamination may be difficult to control however, daily contamination would not be acceptable.

4) Out-of-control surrogate recoveries in the method blank require reanalysis of the method blank.

6.4.3 Documentation

6.4.3.1 Copies of all method blank analyses are stored with the sample analyses raw data. In addition, the raw data for the method blank analyses are stored in the laboratory for an extended period of time. The results for the method blank analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank(s) are noted in the instrument run log. Outliers are discussed in the case narrative.

6.5 QC Check Sample (LCS)

6.5.1 The LCS is used to determine whether a method is in control during sample preparation and analysis. A LCS follows the same protocol as the matrix spike analysis except that the spiking solution is added to a blank matrix (deionized water for water or sea sand for soil/sediments) instead of an actual sample. A Lab Control Sample is prepared with every set of samples extracted at the same time, at a frequency of at least one LCS per 20 samples. It is recommended that all target analytes of concern be spiked into the LCS. A subset of the single-component target analytes can be substituted for the full list if specified in project-specific contracts or workplans per the USACOE Shell document and DoD Quality Systems Manual – Final Version 1, October 2000.

6.5.2 The LCS may fill the requirements for the performance of an initial calibration and continuing calibration verification standard as specified in Sections 9.4.2.1 and 9.4.2.2 in the DoD Quality Systems Manual – Final Version 1, October 2000. The spiking levels vary from 20µg/mL to 30µg/mL.

6.5.3 Criteria

6.5.3.1 At the time of the writing of this SOP, the established control limits are as follows:

TABLE 1
Blank Spike Recovery Limits

Compound	%Recovery Water	%Recovery Soil/Sediment
Phenol	10-101	24-121
2-Chlorophenol	11-117	29-116
1,4-Dichlorobenzene	20-97	27-111
N-Nitroso-di-n-propylamine	40-123	23-123
1,2,4-Trichlorobenzene	22-102	29-114
4-Chloro-3-methylphenol	40-114	32-120
Acenaphthene	49-112	35-118

Compound	%Recovery Water	%Recovery Soil/Sediment
4-Nitrophenol	10-119	10-147
2,4-Dinitrotoluene	41-120	24-121
Pentachlorophenol	10-160	10-158
Pyrene	48-132	27-127

6.5.4 Corrective action

6.5.4.1 Check all peak integrations and sample calculations.

6.5.4.2 If the analyte recoveries still exceed the control limits, the blank spike extract may be reanalyzed.

6.5.4.3 If the analyte recoveries are still out of control, re-extraction of the associated samples is required.

6.6 Matrix Spike/Matrix Spike Duplicate Sample

6.6.1 A sample is chosen from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is not required that a matrix spike analysis be performed with each extraction batch unless the project QAPP requires it. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of Calculate the recovery of each matrix spike compound using the equation below.

$$\%\text{Recovery} = \frac{Spiked\ \ sample\ result - sample\ result}{Spike\ \ added} \times 100$$

6.6.2 Calculate the relative percent difference (RPD) of the recoveries of each compound in the matrix spike and matrix spike duplicate as follows:

$$RPD = \frac{|Matrix\ spike\ recovery\ -\ Matrix\ spike\ duplicate\ recovery|}{1/2(Matrix\ spike\ recovery\ +\ Matrix\ spike\ duplicate\ recovery)} \times 100$$

The vertical bars in the formula above indicate the absolute value of the difference, therefore RPD is always expressed as a positive value.

6.6.3 Criteria

6.6.3.1 At the time of the writing of this SOP, the established control limits are as follows:

TABLE 2
Matrix Spike Recovery and Relative Percent Difference Limits

Compound	%Recovery Water	RPD Water	%Recovery Soil/Sediment	RPD Soil/Sediment
Phenol	33-112	35	41-109	28
2-Chlorophenol	41-115	35	40-106	30
1,4-Dichlorobenzene	33-96	39	34-107	36
N-Nitroso-di-n-propylamine	53-128	38	48-118	28
1,2,4-Trichlorobenzene	43-103	37	40-121	30
4-Chloro-3-methylphenol	49-121	35	55-120	22
Acenaphthene	47-121	39	41-122	42
4-Nitrophenol	38-134	43	23-143	37
2,4-Dinitrotoluene	52-122	38	32-127	25
Pentachlorophenol	60-131	39	20-159	43
Pyrene	52-116	36	25-141	50

6.6.4 Corrective action

6.6.4.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases, a narrative explanation of the condition is required to detail the corrective actions taken.

6.6.4.2 If any analyte recovery exceeds the control limits the following action must be taken:

6.6.4.2.1 If the blank spike analysis yielded recoveries outside the control limits then re-extract and reanalyze all samples extracted with the MS/MSD.

6.6.4.2.2 If the blank spike analysis was in control then recoveries outside the control limits can be attributed to matrix effect and sample results are processed as normal. The report comments or narrative section must include comments about the out-of-control recoveries.

6.6.4.3 Corrective action for RPD values which exceed the control limits follows the corrective action for MS/MSD recoveries. If more than one RPD exceeds the control limit, re-extraction may not be required if it can be demonstrated that the sample is non-homogeneous and all MS/MSD recoveries are within the control limits.

6.7 Surrogate Recovery

6.7.1 Surrogates are chemically similar compounds added to every sample, method blank and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects.

6.7.2 Surrogate compound recoveries are calculated as follow:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:
S_m = Concentration of surrogate measured in extract.
S_a = Concentration of surrogate added.

6.7.3 Criteria

6.7.4 The semivolatile surrogates can be divided into two groups: base/neutral compounds (nitrobenzene-d5, 2-fluorobiphenyl, 1,2-dichlorobenzene-d4, and terphenyl-d14); acid compounds (phenol-d5, 2-fluorophenol, 2,4,6-tribromophenol, and 2-chlorophenol-d4). At the time of the writing of this SOP, the established control limits are follows:

TABLE 3
SURROGATE RECOVERY LIMITS

Compound	%Recovery Water	%Recovery Soil
Nitrobenzene-d5	35-105	35-140
2-Fluorobiphenyl	30-88	35-140
Terphenyl-d14	33-129	35-140
Phenol-d5	10-94	35-140
2-Fluorophenol	10-100	35-140
2,4,6-Tribromophenol	10-123	35-140
2-Chlorophenol-d4	10-120 (advisory)	35-140 (advisory)
1,2-Dichlorobenzene-d4	28-94 (advisory)	35-140 (advisory)

6.7.5 Corrective Action

6.7.5.1 Check calculations for possible error.

6.7.5.2 Check instrument performance, if necessary correct the problem and re-analyze the extract.

6.7.5.3 Some samples may require dilution in order to bring one or more target analytes within the calibration range or to overcome significant matrix interference. This may result in the dilution of the surrogate response to the point that the recoveries can not be measured. If the surrogate recoveries are available from a less-diluted or undiluted aliquot of the sample or sample extract, those recoveries may be used to demonstrate that the surrogates were within the QC limits, and no further action is required. For all package work both the diluted and undiluted analyses will be provided.

6.7.5.4 Re-extraction is not necessary in the case where a sample is chosen for MS/MSD analyses and the same recovery pattern is present in all three analyses. In this case, matrix effects may be assumed and re-extraction is not required for this sample.

6.7.5.5 If only one fraction is being analyzed and surrogates that relate to the other fraction are out-of-control, then re-extraction and reanalysis for the target fraction is not required.

6.7.5.6 All other circumstances require re-extraction and reanalysis of the affected sample(s).

6.7.5.7 Out of control surrogate recoveries in the method blank require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Edition, Method 8270D, "Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS): Capillary Column Technique," Revision 4, January 1998, U.S. EPA.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, 2nd Update, Method 3640A, "Gel-Permeation Cleanup," Revision 1, November 1992, US EPA.

US EPA. Contract Laboratory Program, Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, Document Number OLM04.2, May 1999.

APPENDIX I

Calibration Standard Solution Concentrations, ng/µL

Compound	STD1	STD2	STD3	STD4	STD5	STD6
Phenol	5	10	25	40	60	80
bis(2-Chloroethyl)ether	5	10	25	40	60	80
2-Chlorophenol	5	10	25	40	60	80
1,3-Dichlorobenzene	5	10	25	40	60	80
1,4-Dichlorobenzene	5	10	25	40	60	80
Benzyl Alcohol	5	10	25	40	60	80
1,2-Dichlorobenzene	5	10	25	40	60	80
2-Methylphenol	5	10	25	40	60	80
Bis(2-chloroisopropyl)ether	5	10	25	40	60	80
4-Methylphenol	5	10	25	40	60	80
N-Nitroso-di-n-propylamine	5	10	25	40	60	80
Hexachloroethane	5	10	25	40	60	80
Nitrobenzene	5	10	25	40	60	80
Isophorone	5	10	25	40	60	80
2-Nitrophenol	5	10	25	40	60	80
2,4-Dimethylphenol	5	10	25	40	60	80
Benzoic Acid	5	10	25	40	60	80
bis(2-Chloroethoxy)methane	5	10	25	40	60	80
2,4-Dichlorophenol	5	10	25	40	60	80
1,2,4-Trichlorobenzene	5	10	25	40	60	80
Naphthalene	5	10	25	40	60	80
4-Chloroaniline	5	10	25	40	60	80
Hexachlorobutadiene	5	10	25	40	60	80
4-Chloro-3-methylphenol	5	10	25	40	60	80
2-Methylnaphthalene	5	10	25	40	60	80
Hexachlorocyclopentadiene	5	10	25	40	60	80
2,4,6-Trichlorophenol	5	10	25	40	60	80
2,4,5-Trichlorophenol	5	10	25	40	60	80
2-Chloronaphthalene	5	10	25	40	60	80
2-Nitroaniline	5	10	25	40	60	80
Dimethylphthalate	5	10	25	40	60	80
Acenaphthylene	5	10	25	40	60	80
2,6-Dinitrotoluene	5	10	25	40	60	80
3-Nitroaniline	5	10	25	40	60	80
Acenaphthene	5	10	25	40	60	80
2,4-Dinitrophenol	5	10	25	40	60	80

Compound	STD1	STD2	STD3	STD4	STD5	STD6
4-Nitrophenol	5	10	25	40	60	80
Dibenzofuran	5	10	25	40	60	80
2,4-Dinitrotoluene	5	10	25	40	60	80
Diethylphthalate	5	10	25	40	60	80
4-Chlorophenyl-phenylether	5	10	25	40	60	80
Fluorene	5	10	25	40	60	80
4-Nitroaniline	5	10	25	40	60	80
4,6-Dinitro-2-methylphenol	5	10	25	40	60	80
N-Nitrosodiphenylamine	5	10	25	40	60	80
4-Bromophenyl-phenylether	5	10	25	40	60	80
Hexachlorobenzene	5	10	25	40	60	80
Pentachlorophenol	5	10	25	40	60	80
Phenanthrene	5	10	25	40	60	80
Anthracene	5	10	25	40	60	80
Carbazole	5	10	25	40	60	80
Di-n-butylphthalate	5	10	25	40	60	80
Fluoranthene	5	10	25	40	60	80
Pyrene	5	10	25	40	60	80
Butylbenzylphthalate	5	10	25	40	60	80
3,3'-Dichlorobenzidine	5	10	25	40	60	80
Benzo(a)anthracene	5	10	25	40	60	80
Chrysene	5	10	25	40	60	80
bis(2-Ethylhexyl)phthalate	5	10	25	40	60	80
Di-n-octylphthalate	5	10	25	40	60	80
Benzo(b)fluoranthene	5	10	25	40	60	80
Benzo(k)fluoranthene	5	10	25	40	60	80
Benzo(a)pyrene	5	10	25	40	60	80
Indeno(1,2,3-cd)pyrene	5	10	25	40	60	80
Dibenzo(a,h)anthracene	5	10	25	40	60	80
Benzo(g,h,i)perylene	5	10	25	40	60	80
N-Nitrosodimethylamine	5	10	25	40	60	80
2-Fluorophenol	5	10	25	40	60	80
Aniline	5	10	25	40	60	80
Phenol-d5	5	10	25	40	60	80
2-Chlorophenol-d4	5	10	25	40	60	80
1,2-Dichlorobenzene-d4	5	10	25	40	60	80
Nitrobenzene-d5	5	10	25	40	60	80
2-Fluorobiphenyl	5	10	25	40	60	80
2,4,6-Tribromophenol	5	10	25	40	60	80
1,2-Diphenylhydrazine	5	10	25	40	60	80
Benzidine	5	10	25	40	60	80
Terphenyl-d14	5	10	25	40	60	80

APPENDIX II

DFTPP Tuning Criteria

Mass	Relative Abundance
51	30-80% of mass 198
68	less than 2% of 69
69	present
70	less than 2% of 69
127	25-75% of mass 198
197	less than 1% of 198
198	100%
199	5-9% of mass 198
275	10-30% of mass 198
365	greater than 0.75% of mass 198
441	0-100 % of mass 443
442	40-110% of mass 198
443	15-24% of mass 442

CLP requires that the three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak.

SW 846 states that the following approach has been shown to be useful: the mass spectrum of DFTPP may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of DFTPP. Do not background subtract part of the DFTPP peak. Alternatively, the analyst may use other published tuning criteria.

APPENDIX III

Data Packet Order List

QC Summary
Surrogate Recovery Summary Report
Blank Spike Recovery Report
MS/MSD Recovery Report
Method Blank Summary
GC/MS Instrument Performance Check
Internal Standard Area and RT Summary Report

Sample Data
Target Compound Results (Organics Analysis Data Sheet)
Tentatively Identified Compound (TIC) Results (if required)
Sample Chromatograms, quantitation reports and spectra for all samples

Standards Data
Initial Calibration Summary Report
Chromatograms and quantitation reports for all initial calibration standards
Continuing Calibration Summary Reports
Chromatograms and quantitation reports for all CCV Standards

Raw QC Data
Bar Graph Spectrum, mass listing and chromatogram for every DFTPP injection
Method Blank Data
Target Compound Results (Organics Analysis Data Sheet)
Tentatively Identified Compound (TIC) Results (if required)
Chromatograms, quantitation reports and spectra for all method blanks
Blank Spike Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all blank spikes
Matrix Spike/Matrix Spike Duplicate Data
Target Compound Results (Organics Analysis Data Sheet)
Chromatograms, quantitation reports and spectra for all MS analyses
Control Charts

Bench Sheets
All extraction bench sheets, analysis log book pages, chains of custody, and any other pertinent information

APPENDIX IV

Initial Calibration Criteria

Calibration Check Compound	%RSD Limit
Phenol	≤30%
1,4-Dichlorobenzene	≤30%
2-Nitrophenol	≤30%
2,4-Dichlorophenol	≤30%
Hexachlorobutadiene	≤30%
4-Chloro-3-methylphenol	≤30%
2,4,6-Trichlorophenol	≤30%
Acenaphthene	≤30%
N-Nitrosodiphenylamine	≤30%
Pentachlorophenol	≤30%
Fluoranthene	≤30%
Di-n-octylphthalate	≤30%
Benzo(a)pyrene	≤30%

System Performance Check Compound	Minimum Average RF
N-Nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

APPENDIX V

Continuing Calibration Verification Criteria

Calibration Check Compound	%D Limit
Phenol	≤20%
1,4-Dichlorobenzene	≤20%
2-Nitrophenol	≤20%
2,4-Dichlorophenol	≤20%
Hexachlorobutadiene	≤20%
4-Chloro-3-methylphenol	≤20%
2,4,6-Trichlorophenol	≤20%
Acenaphthene	≤20%
N-Nitrosodiphenylamine	≤20%
Pentachlorophenol	≤20%
Fluoranthene	≤20%
Di-n-octylphthalate	≤20%
Benzo(a)pyrene	≤20%

System Performance Check Compound	Minimum RF
N-Nitroso-di-n-propylamine	0.050
Hexachlorocyclopentadiene	0.050
2,4-Dinitrophenol	0.050
4-Nitrophenol	0.050

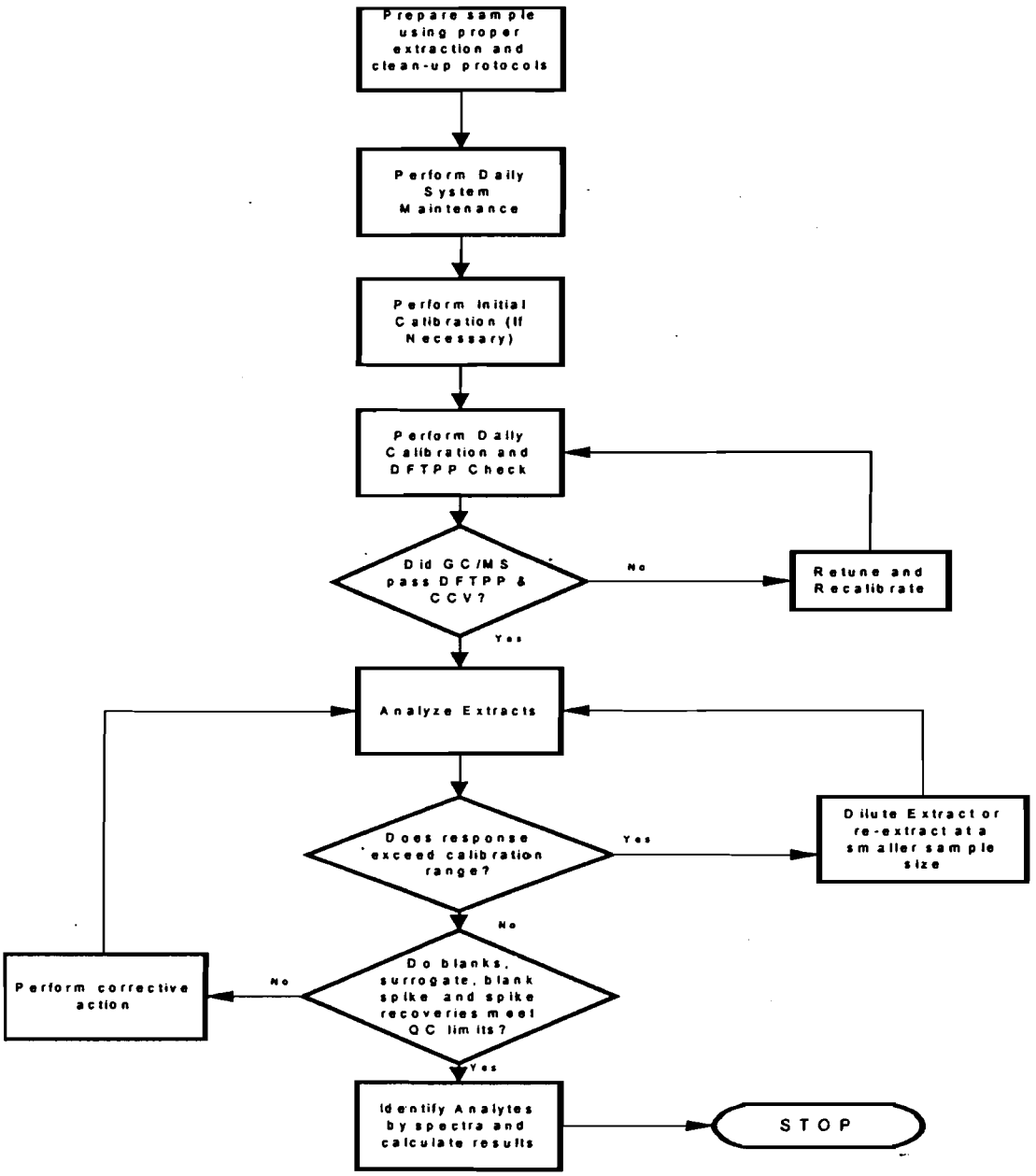
APPENDIX VI

Method Blank Corrective Action Limits

	Control Limits	
Compound	**Water**	**Soil**
Phthalates	RL	RL
All other compounds	1/2 RL	1/2 RL

APPENDIX VII

Method 8270D Flow Chart



APPENDIX VIII

Method 8270D QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
DFTPP Tuning Verification (50 ng)	Mass Abundance 51 30-80% of 198 68 <2% of 69 69 present 70 <2% of 69 127 25-75% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >.75% of 198 441 present but < 443 442 40-110% of 198 443 15-24% of 442	Mass Abundance 51 30-80% of 198 68 <2% of 69 69 present 70 <2% of 69 127 25-75% of 198 197 <1% of 198 198 100% 199 5-9% of 198 275 10-30% of 198 365 >.75% of 198 441 present but < 443 442 40-110% of 198 443 15-24% of 442 Alternatively, any published tuning criteria may be used.	Every 12 hours; CLP Criteria is used as allowed by the method.	Retune instrument to pass criteria.	
Initial Calibration	Minimum of 5 levels, lowest near but above MDL, %RSD for CCC ≤30, All others should be ≤15%. RF for SPCC >0.05.	6 levels, %RSD for CCC ≤30, All others <15%, All RFs >0.010 Alternatively, average RSD must be ≤15%, 40% as an upper limit.	As necessary due to major instrument maintenance or continuing difficulties meeting the CCV requirements.	Re-analysis of out of control standards.	Copies of all raw data, mass calibration, tune, and Form VI.
Initial Calibration Verification		Mid-range standard; concentrations range from 32-64µg/mL. Rec= 70-130% with up to 10 sporadic marginal failures.	Once after initial calibration.	Re-analysis of initial calibration.	
Continuing Calibration Verification	Mid-level standard every 12 hours. %D ≤20% for CCCs SPCCs >0.05. IS Area must be -50% to +100% of the IS in the ICV. RT of IS must be within ±30 seconds of the RT of the IS in the IC.	25 ng/µl standard every 12 hours. %D for CCC ≤20. All RFs >0.010. Average %D must be ≤15%. 40% as an upper limit. IS Area must be -50% to +100% of the IS in the IC. No RT limits for IS in the CCV.	Every 12 hours.	Perform system maintenance, re-analyze CCV standard.	

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Internal Standards	1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 40 ng/uL ea..	1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, Perylene-d12, all @ 20 ng/µl ea. Area must be -50% to +100% of the area of the IS in the CCV. RT must be within 0.5 minutes of RT in CCV.	All standards, blanks, matrix spikes, matrix spike duplicates, blank spikes, SRMs, and sample re-extracts.	Re-analyze any sample which IS areas or RTs are out of range.	Notation in case narrative if applicable.
Method Blank	Analytes must be <MDL except phthalates which must be <5X the MDL.	All analytes <1/2 RL, except phthalates up to RL.	One method blank per 20 samples or each extraction batch of samples, whichever is more frequent.	Re-analyze blank. If still out of control, re-extract the entire batch of samples unless the offending analyte(s) are not detected in the associated samples.	Notation in case narrative if any analytes >1/2 reporting limit are present.
Surrogate Recovery	Nitrobenzene-d5, 2-Fluorobiphenyl, Terphenyl-d14, Phenol-d6, 2-Fluorophenol, & 2,4,6-Tribromophenol. Upper and lower recovery limits determined by 3X standard deviation of at least twenty samples. To be performed at least semi-annually.	Nitrobenzene-d5, 2-Fluorobiphenyl, Terphenyl-d14, Phenol-d6, 2-Fluorophenol, & 2,4,6-Tribromophenol. Upper and lower recovery limits determined by 3X standard deviation of at least twenty samples. To be performed at least semi-annually.	All samples, method blanks, LCSs, LESs.	Re-extraction of the sample is required if any surrogate is out. All surrogates must be in control in the method blank, otherwise all associated samples must be re-extracted.	Notation in case narrative if applicable.
Blank Spike Recovery	MS/MSD Recovery criteria	Empirically derived from five or more blank spikes. QC limit equals 3X standard deviation. See QC database.	One per batch of twenty.	Re-extract batch unless MS/MSD is perfect.	Narrate in case narrative.
MS/MSD Recovery & RPD	Empirically derived from five or more MS/MSD pairs. QC interval equals 3X standard deviation.	Empirically derived from five or more MS/MSD pairs. QC interval equals 3X standard deviation. See QC database.	One per batch of twenty.	Re-extract batch unless blanks spike is perfect or documentable matrix effect is present.	Narrate problems in case narrative.

APPENDIX IX

Appendix IX Calibration Standard Solution Concentrations, ng/µL

Compound	STD1	STD2	STD3	STD4	STD5	STD6
2,6-Dichlorophenol	5	10	25	40	60	80
1,2,4,5-Tetrachlorobenzene	5	10	25	40	60	80
2,3,4,6-Tetrachlorophenol	5	10	25	40	60	80
Dinoseb	5	10	25	40	60	80
Methanesulfonate, methyl	5	10	25	40	60	80
Methanesulfonate, ethyl	5	10	25	40	60	80
Phosphorothiotic Acid	5	10	25	40	60	80
Safrole	5	10	25	40	60	80
Isosafrole, cis & trans	5	10	25	40	60	80
1,4-Naphthoquinone	5	10	25	40	60	80
1,3-Dinitrobenzene	5	10	25	40	60	80
Thionazin	5	10	25	40	60	80
2,4-D, methyl ester	5	10	25	40	60	80
Diallate	5	10	25	40	60	80
Phorate	5	10	25	40	60	80
Dimethoate	5	10	25	40	60	80
Silvex, methyl ester	5	10	25	40	60	80
2,4,5-T, methyl ester	5	10	25	40	60	80
Pronamide	5	10	25	40	60	80
Disulfoton	5	10	25	40	60	80
Methyl parathion	5	10	25	40	60	80
Ethyl parathion	5	10	25	40	60	80
Isodrin	5	10	25	40	60	80
Chlorobenzilate	5	10	25	40	60	80
Kepone	5	10	25	40	60	80
Famphur	5	10	25	40	60	80
3-Methylcholanthrene	5	10	25	40	60	80
Ethanamine,N-methyl-N-nitroso	5	10	25	40	60	80
N-nitrosomorpholine	5	10	25	40	60	80
Butanamine, N-butyl-N-nitroso	5	10	25	40	60	80
2-Naphthalenamine	5	10	25	40	60	80
Diphenylamine	5	10	25	40	60	80
1,3,5-Trinitrobenzene	5	10	25	40	60	80
4-Aminobiphenyl	5	10	25	40	60	80
Methyapyrilene, HCL	5	10	25	40	60	80

Compound	STD1	STD2	STD3	STD4	STD5	STD6
p-Dimethylaminoazobenzene	5	10	25	40	60	80
2-Acetylaminofluorene	5	10	25	40	60	80
7,12-Dimethylbenz(a)anthracene	5	10	25	40	60	80
2-Picoline	5	10	25	40	60	80
Pentachloroethane	5	10	25	40	60	80
Acetophenone	5	10	25	40	60	80
N-Nitrosopyrrolidine	5	10	25	40	60	80
N-Nitrosopiperidine	5	10	25	40	60	80
Hexachloropropene	5	10	25	40	60	80
1,4-Phenylenediamine	5	10	25	40	60	80
Pentachlorobenzene	5	10	25	40	60	80
1-Naphthylamine	5	10	25	40	60	80
5-Nitro-o-toluidine	5	10	25	40	60	80
Pentachloronitrobenzene	5	10	25	40	60	80
N-Nitroquinoline-N-oxide	5	10	25	40	60	80
3,3-Dimethylbenzidine	5	10	25	40	60	80
Hexachlorophene	5	10	25	40	60	80
N-Nitrosodiethylamine	5	10	25	40	60	80
o-Toluidine	5	10	25	40	60	80
Phentermine	5	10	25	40	60	80
Sulfotep	5	10	25	40	60	80
Aramite	5	10	25	40	60	80
Phenacetin	5	10	25	40	60	80

Appendix X

Analytes Amenable to Analysis by Method 8270D

Current 8270D Target Analytes:

Compound	Primary Ion	Secondary Ion(s)
Phenol	94	65, 66
bis(2-Chloroethyl)ether	93	63, 95
2-Chlorophenol	128	64, 130
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
Benzyl Alcohol	108	79, 77
1,2-Dichlorobenzene	146	148, 111
2-Methylphenol	107	108, 77
Bis(2-chloroisopropyl)ether	45	77, 121
4-Methylphenol	107	108, 77
N-Nitroso-di-n-propylamine	70	42, 101
Hexachloroethane	117	201, 199
Nitrobenzene	77	123, 65
Isophorone	82	195, 138
2-Nitrophenol	139	109, 65
2,4-Dimethylphenol	122	107, 121
Benzoic Acid	122	105, 77
bis(2-Chloroethoxy)methane	93	95, 123
2,4-Dichlorophenol	162	164, 98
1,2,4-Trichlorobenzene	180	182, 145
Naphthalene	128	129, 127
4-Chloroaniline	127	129, 65
Hexachlorobutadiene	225	223, 227
4-Chloro-3-methylphenol	107	144, 142
2-Methylnaphthalene	142	141
Hexachlorocyclopentadiene	237	235, 272
2,4,6-Trichlorophenol	196	198, 200
2,4,5-Trichlorophenol	196	198, 97
2-Chloronaphthalene	162	127, 164
2-Nitroaniline	65	92, 138
Dimethylphthalate	163	194, 164
Acenaphthylene	152	151, 153
2,6-Dinitrotoluene	165	63, 89

Current 8270D Target Analytes:

Compound	Primary Ion	Secondary Ion(s)
3-Nitroaniline	138	108, 92
Acenaphthene	154	153, 152
2,4-Dinitrophenol	184	63, 154
4-Nitrophenol	139	109, 65
Dibenzofuran	168	139
2,4-Dinitrotoluene	167	
4-Nitroaniline	138	65, 108
4,6-Dinitro-2-methylphenol	198	51, 105
N-Nitrosodiphenylamine	169	168, 167
4-Bromophenyl-phenylether	248	250, 141
4-Chlorophenyl-phenylether	204	206, 141
Fluorene	166	165, 167
Diethylphthalate	149	177, 150
Hexachlorobenzene	284	142, 249
Pentachlorophenol	266	264, 268
Phenanthrene	178	179, 176
Anthracene	178	176, 179
Carbazole	167	84
Di-n-butylphthalate	149	150
Fluoranthene	202	101
Pyrene	202	200, 203
Butylbenzylphthalate	149	91
3,3'-Dichlorobenzidine	252	254, 126
Benzo(a)anthracene	228	229, 226
Chrysene	228	226, 229
bis(2-Ethylhexyl)phthalate	149	167, 279
Di-n-octylphthalate	149	167, 43
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(a)pyrene	252	253, 125
Indeno(1,2,3-cd)pyrene	276	138, 227
Dibenzo(a,h)anthracene	278	139, 279
Benzo(g,h,i)perylene	276	138, 277
N-Nitrosodimethylamine	74	42
2-Fluorophenol	112	64
Aniline	93	66, 65

Current 8270D Target Analytes:

Compound	Primary Ion	Secondary Ion(s)
Phenol-d5	99	42, 71
2-Chlorophenol-d4	132	68
1,2-Dichlorobenzene-d4	152	115
Nitrobenzene-d5	82	128, 54
2-Fluorobiphenyl	172	171
2,4,6-Tribromophenol	330	332, 141
1,2-Diphenylhydrazine	77	182, 105
Benzidine	184	92, 185
Terphenyl-d14	244	122, 212

Current Appendix IX Analytes:

Compound	Primary Ion	Secondary Ion(s)
2,6-Dichlorophenol	162	164, 63
1,2,4,5-Tetrachlorobenzene	216	214, 218
2,3,4,6-Tetrachlorophenol	230	232, 234
Dinoseb	211	163, 117
Methanesulfonate, methyl	80	79, 95
Methanesulfonate, ethyl	109	97
Phosphorothiotic Acid	97	121, 198
Safrole	162	104, 131
Isosafrole, cis & trans	162	104, 131
1,4-Naphthoquinone	158	102, 104
1,3-Dinitrobenzene	168	76, 122
Thionazin	97	96, 107
2,4-D, methyl ester	199	175, 234
Diallate	86	234, 236
Phorate	260	231, 121
Dimethoate	87	93, 125
Silvex, methyl ester	196	225, 282
2,4,5-T, methyl ester	233	235, 268
Pronamide	173	175, 255
Disulfoton	88	274, 142
Methyl parathion	263	125, 109
Ethyl parathion	291	235, 186
Isodrin	193	195, 230

Current Appendix IX Analytes:

Compound	Primary Ion	Secondary Ion(s)
Chlorobenzilate	139	251, 253
Kepone	272	274, 355
Famphur	218	125, 217
3-Methylcholanthrene	268	252, 253
Ethanamine,N-methyl-N-nitroso	42	88, 43
N-nitrosomorpholine	56	86, 116
Butanamine, N-butyl-N-nitroso	84	41, 57
2-Naphthalenamine	143	144, 115
Diphenylamine	169	168, 167
1,3,5-Trinitrobenzene	213	75, 74
4-Aminobiphenyl	169	168, 170
Methyapyrilene, HCL	58	97, 191
p-Dimethylaminoazobenzene	120	225, 226
2-Acetylaminofluorene	181	180, 223
7,12-Dimethylbenz(a)anthracene	256	241, 239
2-Picoline	93	92, 78
Pentachloroethane	167	165, 119
Acetophenone	105	77, 51
N-Nitrosopyrrolidine	100	41, 42
N-Nitrosopiperidine	42	114, 55
Hexachloropropene	213	215, 211
1,4-Phenylenediamine	108	80, 81
Pentachlorobenzene	250	252, 248
1-Naphthylamine	143	144, 115
5-Nitro-o-toluidine	152	77, 106
Pentachloronitrobenzene	237	214, 295
N-Nitroquinoline-N-oxide	190	160, 89
3,3-Dimethylbenzidine	212	213, 211
Hexachlorophene	196	198, 209
N-Nitrosodiethylamine	74	42
o-Toluidine	106	107
Phentermine	58	91, 134
Sulfotep	97	322, 202
Aramite	135	193, 208
Phenacetin	108	109, 179

APPENDIX XI

Semivolatile Internal Standards With Corresponding Analytes Assigned For Quantitation

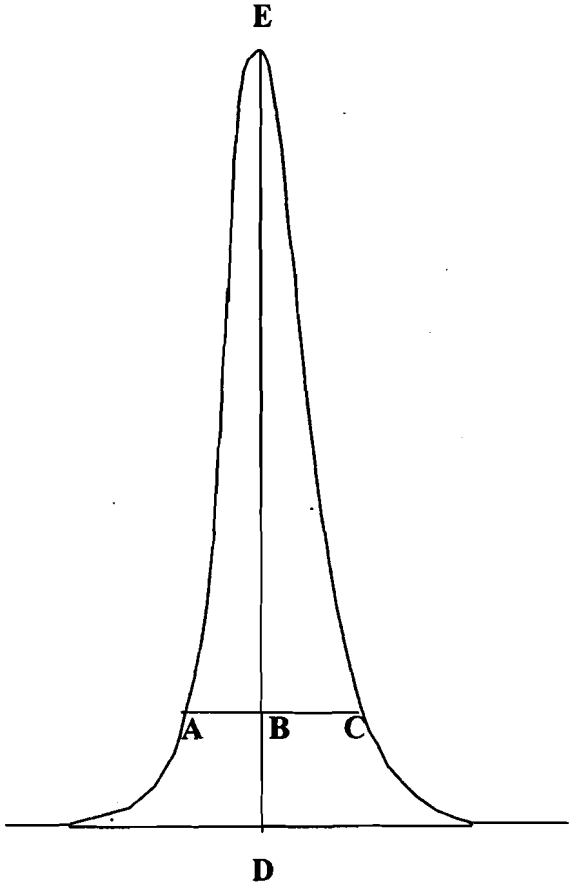
1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
Phenol	Nitrobenzene	Hexachlorocyclopentadiene
Bis(2-Chloroethyl)ether	Isophorone	2,4,6-Trichlorophenol
2-Chlorophenol	2-Nitrophenol	2,4,5-Trichlorophenol
1,3-Dichlorobenzene	2,4-Dimethylphenol	2-Chloronaphthalene
1,4-Dichlorobenzene	Benzoic Acid	2-Nitroaniline
Benzyl Alcohol	bis(2-Chloroethoxy)methane	Dimethylphthalate
1,2-Dichlorobenzene	2,4-Dichlorophenol	Acenaphthylene
2-Methylphenol	1,2,4-Trichlorobenzene	2,6-Dinitrotoluene
Bis(2-chloroisopropyl)ether	Naphthalene	3-Nitroaniline
4-Methylphenol	4-Chloroaniline	Acenaphthene
N-Nitroso-di-n-propylamine	Hexachlorobutadiene	2,4-Dinitrophenol
Hexachloroethane	4-Chloro-3-methylphenol	4-Nitrophenol
Aniline	2-Methylnaphthalene	Dibenzofuran
Pyridine	Nitrobenzene-d5	2,4-Dinitrotoluene
Phenol-d5	2,6-Dichlorophenol	4-Nitroaniline
2-Chlorophenol-d4	Safrole	Fluorene
1,2-Dichlorobenzene-d4	Isosafrole, cis & trans	4-Chlorophenyl-phenylether
N-Nitrosodimethylamine	1,4-Naphthoquinone	Diethylphthalate
2-Fluorophenol	Butanamine, N-methyl-N-nitroso	1,2,4,5-Tetrachlorobenzene
Methanesulfonate methyl	Hexachloropropene	2,3,4,6-Tetrachlorophenol
Methanesulfonate ethyl	1,4-phenylenediamine	1,3-Dinitrobenzene
Phosphorothiotic acid	Phentermine	Thionazin
Ethanamine, N-methyl-N-nitroso		2,4-D methyl ester
N-Nitrosomorpholine		Diallate
2-Picoline		Phorate
Pentachloroethane		Dimethoate
Acetophenone		Silvex methyl ester
N-Nitrosopyrrolidine		2,4,5-T methyl ester
N-Nitrosopiperidine		2-Naphthaleneamine

1,4-Dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
N-Nitrosodiethylamine O-Toluidine		Pentachlorobenzene 1-Naphthylamine 5-Nitro-o-toluidine Pentachloronitrobenzene Sulfotep Aramite

Phenanthrene-d10	Chrysene-d12	Perylene-d12
4,6-Dinitro-2-methylphenol N-Nitrosodiphenylamine 4-Bromophenyl-phenylether Hexachlorobenzene Pentachlorophenol Phenanthrene Anthracene Carbazole Di-n-butylphthalate Fluoranthene 2,4,6-Tribromophenol 1,2-Diphenylhydrazine Dinoseb Pronamide Disulfoton Methyl parathion Ethyl parathion Isodrin Diphenylamine 1,3,5-Trinitrobenzene 4-Aminobiphenyl Methapyrilene, HCL N-Nitroquinoline-n-oxide 3,3-Dimethylbenzidine Phenacetin	Pyrene Butylbenzylphthalate 3,3'-Dichlorobenzidine Benzo(a)anthracene Chrysene bis(2-Ethylhexyl)phthalate Benzidine Terphenyl-d14 Chlorobenzilate Kepone Famphur P-Dimethylaminoazobenzene 2-Acetylaminofluorene 7,12- Dimethylbenz(a)anthracene Hexachlorophene	Di-n-octylphthalate Benzo(b)fluoranthene Benzo(k)fluoranthene Benzo(a)pyrene Indeno(1,2,3-cd)pyrene Dibenzo(a,h)anthracene Benzo(g,h,i)perylene 3-Methylcholanthrene

Appendix XII

Calculation of Peak Tailing Factors



Peak Tailing Factor = BC/AB

Sample calculation: Peak Height = DE =100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm
AB = 11mm
BC = 12mm
Tailing Factor = 12/11 = 1.1

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8303

Determination of Picric and Picramic Acids

Revision history:

Number	Date
1	10/11/90
2	10/11/93
3	01/28/98
4	10/27/98
5	05/09/02

Revised by: _____ Date: 5/10/02
Chad Christianson, Organic Chemist

Approved by: Harry Romberg Date: 5-13-02
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 5-13-02
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 Assigned to: Tetc

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the analysis of ordnance degradation products by high performance liquid chromatography. This method employs the use of a CN (cyano) reverse phase column for the primary column, and a C18 (octadecyl) reverse phase column for confirmation. Target analyte concentrations are measured at 254 nm using a UV detector. The separation employed utilizes an ion-pairing technique.

Compound	Acronym
2,4,6-Trinitrophenol	Picric Acid
2-Amino-4,6-dinitrophenol	Picramic Acid

1.1.2 This method is restricted to use by, or under the supervision of, analysts experienced in the use of high pressure liquid chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.1.3 In order to attain lab-wide consistency among staff members for decision-making processes with regard to laboratory anomalies, several common items have been addressed in this SOP. Any occurrences which are not covered in this SOP should be discussed with the supervisor, prior to implementing a solution.

1.1.4 One example is the determination of potential carry-over in sample analyses. Any samples analyzed subsequent to a high level sample (which is defined by yielding one or more target analytes above the calibration range) should be thoroughly examined for potential carry-over of the same target analyte(s). Corrective action in the form of reanalysis for possible carry-over should be performed and documented in the narrative.

1.1.5 Another example is the review of all sample chromatograms for analytes which may not appear on the sample quantitation report due to data system error or retention time shift. All peaks should be examined and evaluated based on the retention times and sample concentration in order to prevent reporting false negatives.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ±2°C. According to guidelines listed in SW-846 for the extraction of semivolatile compounds, water samples must be extracted within 7 days of

collection, and soil samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.
CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CF	Calibration factor. The ratio of peak response to nanograms injected. This term is defined in the same way in both the CLP contract and SW-846.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	An instrument blank is solvent containing the method surrogates and is injected into the instrument to monitor for carryover between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined at Laucks.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW-846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts, and terminated with a standard injection. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the chromatography column.
RT Window	Retention time window. The ± value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

Primary Column:	PAH C18 (octadecyl) reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm particle size, (Supelcosil PAH-C18 or equivalent).
Secondary Column:	CN (cyano) reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm particle size, (Supelcosil LC-CN or equivalent).
Mobile Phase:	PIC A Reagent for the PAH (or C18) column - (tetrabutylammonium phosphate ion-pairing agent purchased from Waters, part # WAT085101.) Methanol - (EM Science brand high purity solvent or equivalent) Acetonitrile with 0.1% phosphoric acid in water for the CN column Reagent water - (Modulab Polisher HPLC grade water or equivalent).
UV Detector:	254 nm
HPLC System:	Rainin HPLC system - HPXL solvent delivery system capable of achieving 4000 psi or equivalent. 50 µL sample loop. Knauer variable wavelength UV detector or equivalent. Dynamax automatic sample injector or equivalent. Digital integrator: EZChrom or equivalent. Waters System - Waters 712 WISP Sample Processor or equivalent. 50 µL sample loop or equivalent. Waters 486 tunable absorbance detector or equivalent. Waters 600E Multisolvent delivery system or equivalent. Digital Integrator: EZChrom.

2.1.1 Column Temperature Control:

2.1.2 Column temperature can be controlled through the use of a column heater surrounding the column that is maintained at a temperature of 25°C. However, this is not required, and is currently not used.

2.2 Standards

2.2.1 Ion-Pairing Agent (confirmation only)

2.2.1.1 Prepare 1000 mL 1:1 methanol/HPLC grade DIW. Add contents of 1 bottle PIC A Reagent (tetrabutylammonium phosphate). Stir 5 minutes, then filter using a solvent resistant 0.22 micron filter (Millipore GVWP-04700 filters or equivalent).

2.2.2 Individual Target Analyte Stock Solution

2.2.2.1 The picramic acid, picric acid and 4,6-dinitro-o-cresol (surrogate) standards are purchased individually from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The standard solutions come in 1 mL ampules at 1000 µg/mL in 1:1 methanol/acetonitrile.

2.2.3 IBLK Working Solution

2.2.3.1 The working instrument blank solution contains the surrogate at a concentration of 4.0 µg/mL. Prepare this solution by adding 100 µL of the 1000 µg/mL surrogate stock solution to a 25 mL volumetric flask. Dilute to volume with 1:1 methanol/reagent grade water.

2.2.4 Working Calibration Standards

2.2.4.1 Prepare the working calibration standards in the following manner. All solutions are prepared in a 1:1 methanol/water mixture.

Standard	Source Solution	Amount Added	Final Volume (mL)	Final Conc. (µg/mL)
Standard #5	A,B,C	250 µL	10	25
Standard #4	D	5 mL	10	12.5
Standard #3	D	2 mL	10	5
Standard #2	F	2 mL	10	1
Standard #1	F	1 mL	10	0.5

Source Solutions	Concentration (µg/mL)
A = Picramic Acid	1000
B = Picric Acid	1000
C = 4,6-Dinitro-o-cresol	1000
D = Standard 5	25
E = Standard 4	12.5
F = Standard 3	5

2.2.4.2 Note: Store all standard and surrogate solutions in amber glass vials with screw caps and Teflon-lined septa. Minimize headspace in these vials and store them in a refrigerator kept at 4°C ±2°C. Allow standards to come to room temperature prior to use.

2.2.4.3 Stock solutions may be used for up to one year, and working solutions are good for at least six months, or the expiration date of the parent standard, whichever is sooner.

2.2.4.4 All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation standards.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made (e.g. new column) or whenever the chromatographic method is modified.

4.4.3 In some cases, the retention time window may be modified in order to take into account any pattern shifting. This shift is acknowledged by observation of the surrogate peak behavior and the surrounding CCV standards. If retention time shift has occurred and the possibility of mis-identifying peaks exists, then the sample is reanalyzed bracketed by in-control CCV standards.

4.4.4 See the Laucks SOP on determination of retention time windows for more specifics on their determination and use.

4.5 Initial Multi-Point Calibration

4.5.1 Analyze standard solutions (including the surrogate solution) using at least 5 different concentration levels. The lowest concentration standard will determine the reporting limit. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. Laucks routinely uses standard concentrations of 0.5, 1.0, 5.0, 12.5, and 25.0 µg/mL. Criteria for evaluating these standards are detailed in Section 6.

4.6 Initial Calibration Verification

4.6.1 At the beginning of an analysis sequence, analyze the mid-point calibration standard. The computed calibration factor (CF) or concentration measurement must meet the criteria detailed in Section 6.

4.6.2 The retention time windows for compound identification are updated using the retention times for each target analyte in the ICV standard as the center of the window and the previously determined retention time window half-width to establish the retention time range to be used for compound identification.

4.6.3 The retention time windows for compound identification are updated every 24 hours of acquisition time from the ICV standard as the center of the window to a neighboring CCV. This neighboring CCV is determined to be a more accurate representation of peak behavior based on the surrogate, spiked extracts and surrounding standards.

4.7 Instrument Blank

4.7.1 After the analysis of Standard 5, an instrument blank (IBLK) is analyzed. This is to verify that there is no carryover between sample injections. Evaluation criteria are detailed in Section 6.

4.7.2 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK. This IBLK analysis is used only to make a judgment as to the possibility of carryover into the sample analysis immediately following the IBLK. Evaluation criteria are detailed in Section 6.

4.8 Continuing Calibration Verification

4.8.1 A mid-range calibration is analyzed after every ten injections. In addition, this standard must be the last injection made in the analysis sequence. Evaluation criteria are detailed in Section 6.

4.9 Sample Analysis

4.9.1 Analysis sequence

See Appendix II for a detailed analysis injection sequence.

4.9.2 Compound Identification

4.9.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its retention time window on a second column. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria detailed in Section VI are strictly adhered to.

4.9.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes. If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

4.9.3 Compound Quantification

4.9.3.1 Target compound concentrations are calculated using the following equations:

4.9.3.1.1 Aqueous samples

The external standard equation, as expressed in SW-846 is

$$Concentration(\mu g / L) = \frac{As \times Vt \times D}{\overline{CFs} \times Vi \times Vs}$$

where:

As	= Area (or height) of the peak for the analyte in the sample.
V_i	= Volume of extract injected, µL.
D	= Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if an extract is diluted 10 µL to 1000 µL and subsequently diluted an additional 10 µL to 1000 µL, the expression is: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
$\overline{CFs}$	= Mean calibration factor from the initial calibration.
V_t	= Volume of total extract, µL.
V_s	= Volume of aqueous sample extracted or purged in mL. If units of liters are used for this term, multiply the results by 1000.

4.9.3.1.2 To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/L * 0.001

4.9.3.2 Non-aqueous samples

4.9.3.2.1 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Concentration(\mu g / kg) = \frac{Vt \times As \times D}{\overline{CF} \times Vi \times (Ws)}$$

where:

W_s = Weight of sample extracted. Either the wet weight or dry weight may by used, depending upon the specific application of the data.

4.9.3.2.2 One or more analytes may require manual integration on the data system quantitation reports. The manual integrations are flagged, initialed and dated by the analyst. A list of the manual integration flags are detailed below.

M = Manual integration due to irregular peak shape.
MS = Manual integration due to split peak.
MR = Manual integration due to retention time shift.
MI = Manual integration of correct isomer or peak.
MT = Manual integration due to peak tailing.
MB = Manual integration due to irregular baseline.

4.9.3.3 If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

5. Reports

5.1 Data Packet Organization

See Appendix III for a list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Sample Result Reports

5.4 Data Qualifying Flags

5.4.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U:	The analyte of interest was not detected, to the limit of detection indicated.
B:	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.

J:	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D:	The value reported is derived from the analysis of a diluted sample or sample extract.
E:	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
X:	The sample has been analyzed at several dilutions. The value reported has been determined to be the most appropriate quantitative value.

6. Quality Control

See the Requirements and Corrective Actions table in Appendix IV for additional information.

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 The initial calibration is evaluated by calculating the %RSD of the calibration factors from the five linearity standards.

6.1.1.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

6.1.1.3 This %RSD method assumes a linear response with the calibration curve passing through the origin.

6.1.1.4 The calculated CFs are tabulated and the %RSDs calculated. All compounds must have a %RSD of 20% to meet the method criteria.

6.1.2 Corrective action

6.1.2.1 If the %RSD criteria of 20% are not met, the out of control standard should be reanalyzed. If the curve is still out of control, determine the cause of failure and correct.

Recalibrate the instrument and reanalyze any samples associated with the out of control curve.

6.2 Initial Calibration Verification

6.2.1 Criteria

6.2.1.1 Using the average CF calculation technique, compute the CFs for each compound. The calibration factors for the ICV standard are compared to the mean CFs for the initial multi-point calibration. The percent difference for these calibration factors is calculated as follows:

$$\%Difference = \frac{CFi - CFa}{CFa} \times 100$$

where:

CF_a = Mean CF from the initial multi-point calibration
CF_i = Calibration Factor from the calibration verification standard.

6.2.1.2 The %D cannot exceed 15% for any target analytes or surrogates.

6.2.1.3 The mid-point for the RT window of each compound is updated using the ICV RTs.

6.2.2 Corrective action

6.2.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Determine the cause of the ICV failure and correct. Reanalyze the ICV and if it is still out of control, a new calibration curve must be analyzed.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 A CCV standard is analyzed singly for every 10 injections, and after the last sample of the sequence. The CF for each compound is calculated and the percent difference is calculated as shown above.

6.3.1.2 The %D results cannot exceed 15% for any target analytes or surrogates.

6.3.1.3 The retention times for all target analytes must fall within the RT windows established by the ICV.

6.3.2 Corrective action

6.3.2.1 Determine the cause of failure and correct. Reanalyze the calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the RL in the associated samples.

6.4 Instrument Blank

6.4.1 Criteria

6.4.1.1 There must be no target analyte levels above ½ the reporting limit in the initial IBLK.

6.4.2 Corrective action

6.4.2.1 If the initial IBLK contains measurable levels of target analytes, the system is out of control. The source of contamination must be identified and corrected.

6.4.2.2 IBLKs are used to monitor for possible carryover in high concentration extracts. Those IBLKs optionally placed into the sequence following suspected high concentration extracts are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be reanalyzed if there is a detectable amount of the analyte found in the IBLK.

6.5 Method Blank

6.5.1 Criteria

6.5.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or at least one blank for every 20 samples, whichever is more frequent. Analytes of interest should not be detected at levels greater than ½ the current reporting limit in the method blank sample. If any analytes are detected above the reporting limit, corrective action must be taken.

6.5.2 Corrective action

6.5.2.1 Reanalyze the blank and check calculations. If it is still out of control, re-extract the entire batch of samples unless the analyte(s) present in the method blank are not present

in the associated samples. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

6.6 Blank Spike or QC Check Sample (LCS)

6.6.1 Criteria

6.6.1.1 A blank spike follows the same protocol as the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a blank spike. At the time this SOP was written the blank spike percent recovery control limits were between 40 and 160 for both compounds. These control limits are updated periodically.

6.6.2 Corrective action

6.6.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re-extraction and reanalysis would be triggered by any analytes falling outside of control limits in the blank spike sample unless all sample surrogate recoveries and MS/MSD spike recoveries are in control.

6.7 Matrix Spike

6.7.1 Criteria

6.7.1.1 A sample is either chosen at random or is designated by the client from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This frequency may be changed on a project specific basis. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.7.1.2 At the time this SOP was written the matrix spike percent recovery control limits were between 48 and 152 for picric acid, and between 60 and 128 for picramic acid. These control limits are updated periodically.

6.7.2 Corrective action

6.7.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.8 Matrix Spike Duplicate

6.8.1 Criteria

6.8.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} \times 100$$

where:

S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.8.1.2 At the time this SOP was written the matrix spike duplicate percent recovery control limits were between 48 and 152 for picric acid, and between 60 and 128 for picramic acid. These control limits are updated periodically.

6.8.2 Corrective action

If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. At the time this SOP was written the RPD percent control limits were 50. These control limits are updated periodically.

6.9 Surrogate Recovery

6.9.1 Criteria

6.9.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:

S_m = Concentration of surrogate measured in sample.
S_a = Concentration of surrogate added.

6.9.1.2 At the time this SOP was written the surrogate percent recovery control limits were between 48 and 152 for picric acid, and between 50 and 150.

6.9.2 Corrective Action

6.9.2.1 Reanalyze the sample. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recoveries since non-GC/MS methods cannot separate co-eluting interferents. In instances where high surrogate recoveries are attributable to matrix effects, no corrective action is taken. However, if elevated surrogate recoveries are attributable to preparation error, re-extraction and reanalysis is performed.

6.9.2.2 Check calculations for possible error. Re-extract the sample if surrogate recovery is less than the lower control limit. If a poor injection is suspected, reanalyze the sample. A low surrogate recovery in the re-extract is evidence of matrix interference.

6.9.2.3 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8000B, "Gas Chromatography," Revision 2, December 1996, U.S.E.P.A.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, 3rd Update, Method 8330, "Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)," Revision 0, September 1994, U.S.E.P.A.

40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984, U.S.E.P.A.

APPENDIX I

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
Picric Acid	0.5	1.0	5.0	12.5	25.0
Picramic Acid	0.5	1.0	5.0	12.5	25.0
Surrogate					
4,6-Dinitro-o-cresol	0.5	1.0	5.0	12.5	25.0

Surrogate Stock Solution

Compound	Concentration
4,6-Dinitro-o-cresol	1000 µg/mL

IBLK Solution

Compound	Concentration
4,6-Dinitro-o-cresol	4.0 µg/mL

APPENDIX II

Analysis Sequence

Injection	Sample
1	Standard #1
2	Standard #2
3	Standard #3 (CCV Standard)
4	Standard #4
5	Standard #5
6	IBLK
7	ICV
	injections (8-16)
17	IBLK
18	CCV Standard
19	injections (19-27)
28	BLK
29	CCV Standard
	Sample injections (total of 10 injections between CCVs)
last	IBLK
last	CCV Standard

APPENDIX III

Data Packet Organization List

I. QC SUMMARY

Surrogate Recovery Summary Report
Blank Spike Report
MS/MSD Report
Method Blank Summary

II. SAMPLE DATA:

Organic Analysis Data Sheet
Sample Confirmation Worksheet
Chromatograms
Quantitation Report

III. STANDARD DATA:

Linearity Report
ICAL Data
ICAL Response, ICAL std concentrations
ICV reports
CCV reports
Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

Method Blank
Chromatograms
Quantitation Report

Blank Spike
Chromatograms
Quantitation Report

Matrix Spike
Chromatograms
Quantitation Report

Matrix Spike Duplicate
Chromatograms
Quantitation Report

V. Bench Sheets

SDG Report
Extraction Bench Sheets
Injection Sequence (logbook copy)
Miscellaneous Work Sheets. (%TS, calcs, HTVRs)
Standards Logs

VI. Reject Data:

Data not used to support reported sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Picric/Picramic Requirements and Corrective Actions

QA Element	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Minimum of 5 levels. Must demonstrate an RSD of <20%.	As necessary due to major instrument maintenance or difficulties meeting the CCV requirements.	Reanalyze out of control standard. If still out, determine cause of curve failure and correct. Reanalyze curve and any samples analyzed against curve.	Target forms and raw data.
Initial Calibration Verification	+/- 15% of the initial calibration response factor.	Every 24 hrs. or at the beginning of an analytical sequence, which ever is more frequent, or as necessary.	Determine cause of ICV failure and correct. Reanalyze ICV and if out of control a new calibration curve must be analyzed.	Target form and raw data.
Continuing Calibration Verification	+/- 15% of the initial calibration response factor.	Every 10 injections and at the end of an analytical sequence.	Determine cause of failure and correct. Re-analyze calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit.	Target form and raw data.
Instrument Blank	Analyze instrument blank after samples when high levels of matrix are suspected. Analyze instrument blank before the CCV standard. No target analytes present in the blank > ½ the RL. Surrogate must be within control limit.	Analyze instrument blank after samples when high levels of matrix are suspected. Analyze instrument blank before the CCV standard.	Reanalyze any samples with suspected carryover.	Raw data
Method Blank	All analytes must be less than ½ the reporting limit	One method blank per 20 samples or each extraction batch, whichever is more frequent.	Reanalyze blank. If still out of control, re-extract the entire batch of samples unless the detected analyte(s) are not present in the associated samples.	Method Blank Summary and raw data. Narrative comment when necessary.

QA Element	Laucks Criterion	Frequency	Corrective Action	Documentation
Surrogate Recovery	Current control limits are stored in QC database. All surrogate recoveries must fall within the generated limits. If laboratory limits are not established due to the implementation of a new surrogate , the default limits of 70% - 130% are used until enough data points are collected to generate new limits.	All samples, method blanks, and QC samples.	Re-extract if surrogate recovery is < the lower control limit. If a poor injection is suspected re-analyze the sample.	Target Surrogate Summary form or special test results. Narrative comment when necessary.
MS/MSD	Current control limits are stored in the QC database.	1 MS/MSD pair per 20 samples or every extraction batch which ever is more frequent.	If MS/MSD recoveries are out of control, no further action is taken if blank spike recoveries are in control.	MS/MSD report and raw data. Narrative comment when necessary.
Blank Spike	Current control limits are stored in QC database. All recoveries and RPDs must fall within the generated limits.	1 per 20 samples or every extraction batch which ever is more frequent.	Re-extract if analyte recoveries are outside of control limits.	Blank Spike report and raw data. Narrative comment when necessary.

APPENDIX V

Elution Order of Target Analytes

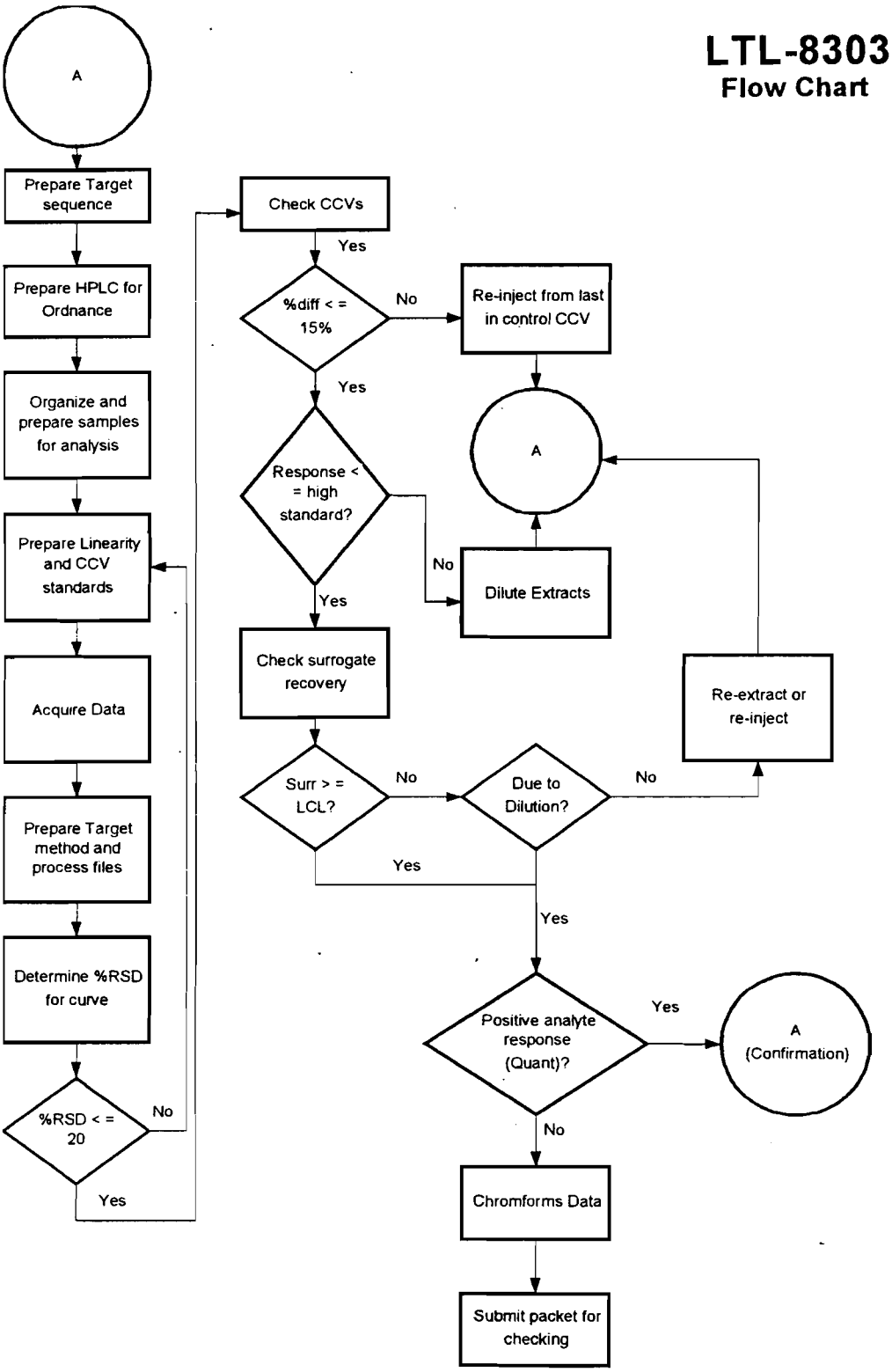
Compound	PAH - C18 Column
Picric Acid	3
Picramic Acid	1
Surrogate	
4,6-Dinitro-o-cresol	2

Compound	CN Column
Picric Acid	1
Picramic Acid	3
Surrogate	
4,6-Dinitro-o-cresol	2

APPENDIX VI

LTL 8303 Flow Chart

LTL-8303
Flow Chart



LAUCKS TESTING LABORATORIES INC.

Seattle, Washington

SOP #:LTL-8330

Determination of Nitroaromatics and Nitramines by SW-846 Method 8330A & 8332

Revision history:

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8	10/28/98
9	01/25/99
10	03/16/00
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Chad Christianson, Organic Chemist

Approved by: Harry Romberg Date: 5-13-02
Harry Romberg, QA Officer

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used for the trace analysis of ordnance compounds in water, soil, and sediment samples by high performance liquid chromatography. The concentrated water sample extracts are diluted 1:1 (v/v) with reagent grade water prior to analysis. The sample extracts are analyzed using a C18 (octadecyl) reverse phase column, and target analyte concentrations are measured at either 254 nm or 210 nm using a UV detector. All positive measurements observed on the C18 column are confirmed by a second analysis which uses a CN (cyano) column (with a C8 column in series for the 6 additional compounds).

1.1.2 The C18 column is considered the primary column and is used for quantitation of all target analytes.

1.1.3 This method is used to determine part per billion levels of the ordnance analytes listed below:

Compound	Acronym
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine	HMX
Hexahydro-1,3,5-trinitro-1,3,5-triazine	RDX
1,3,5-Trinitrobenzene	1,3,5-TNB
1,3-Dinitrobenzene	1,3-DNB
Methyl-2,4,6-trinitrophenylnitramine	Tetryl
Nitrobenzene	NB
2,4,6-Trinitrotoluene	2,4,6-TNT
4-Amino-2,6-dinitrotoluene	4-Am-DNT
2-Amino-4,6-dinitrotoluene	2-Am-DNT
2,4-Dinitrotoluene	2,4-DNT
2,6-Dinitrotoluene	2,6-DNT
2-Nitrotoluene	2-NT
3-Nitrotoluene	3-NT
4-Nitrotoluene	4-NT

6 Additional Compounds
(Separate Analysis at 254 nm)

Compound	Acronym
2,4-Diamino-6-nitrotoluene	2,4-DA-6-NT
2,6-Diamino-4-nitrotoluene	2,6-DA-4-NT
3,5-Dinitroaniline	3,5-DNA
1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine	MNX
1,3,5-Trinitroso-1,3,5-hexahydrotriazine	TNX
2,2',6,6'-Tetranitro-4,4'-azoxytoluene	4,4'-TN-AZOXY

PETN/NG Compounds
(Separate Analysis at 210 nm)

Compound	Acronym
Nitroglycerin	NG
Pentaerythritoltetranitrate	PETN

1.1.4 Aqueous samples of higher concentration can be directly analyzed by diluting 1:1 (v/v) with methanol or acetonitrile, filtering, separating on a C18 reverse phase column, and determined at either 254 nm or 210 nm, and confirmed on a CN column.

1.1.5 This method is restricted to use by, or under the supervision of, analysts experienced in the use of high pressure liquid chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and/or data interpretation.

1.2 Method Deviations & Comments

1.2.1 The following items represent deviations from the published version of method SW 8330 and SW 8332 which are followed as standard operating procedure in the performance of this method at Laucks.

1. Single injections of calibration standards are analyzed rather than triplicate injections in a random order. It has been determined at Laucks that single injections of these standards yield acceptable calibration and linearity data as evidenced by the calculated percent RSDs used to evaluate the initial calibration data.

2. Method SW 8330 and SW 8332 specifies that all analyses are to be performed using a mobile phase which consists of a 50:50 mixture of

methanol/water under isocratic conditions. Laucks employs a gradient elution program (detailed in Section IV) in order to improve the separation of the target analytes on the CN-C8 columns.

3. According to Method 8330 and 8332, all working standards are to be prepared daily in a methanol/calcium chloride solution. The practice at Laucks for 8330/8332 and the 6 additional compounds is to prepare working standards using acetonitrile and diluting 50/50 with water just prior to analysis. NG/PETN are prepared in a 50/50 mixture of acetonitrile and water. These working solutions have been demonstrated to be stable for at least 6 months. The stability of target analyte responses in the working solutions in use (especially that of tetryl) is used to determine whether new solutions should be prepared.

4. Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. Laucks has experienced erratic recovery of tetryl from spiked sample extracts.

5. Currently, the confirmation column will not demonstrate full separation for the ordnance compounds. The following target analytes will co-elute: 2-Nitrotoluene, 3-Nitrotoluene, and 4-Nitrotoluene. As well as 4-Amino-2,6-dinitrotoluene and 2-Amino-4,6-dinitrotoluene. Therefore, positive confirmation can not be made when these peaks have been tentatively identified on the primary column.

6. At the time of this writing, analysis of the 6 additional compounds (not including PETN/NG) combined with the 14 ordnance compounds results in the separation of all compounds on the primary column (C18).

7. Although the compounds NG and PETN are not listed in SW 846 Method 8330/8332, Laucks has found that adequate recovery of these compounds can be achieved using this method by modifying the wavelength from 254 nm to 210 nm.

1.2.2 In order to attain lab-wide consistency among staff members for decision-making processes with regard to laboratory anomalies, several common items have been addressed in this SOP. Any occurrences which are not covered in this SOP should be discussed with the supervisor, prior to implementing a solution.

1.2.3 One example is the determination of potential carry-over in sample analyses. Any samples analyzed subsequent to a high level sample (which is defined by yielding one or more target analytes above the calibration range) should be thoroughly examined for potential carry-over of

the same target analyte(s). Corrective action in the form of reanalysis for possible carry-over should be performed and documented in the narrative, if required.

1.2.4 Another example is the review of all sample chromatograms for analytes which may not show up on the sample quantitation report due to data system error or retention time shift. All peaks should be examined and evaluated based on the retention times and sample concentration in order to prevent reporting false negatives.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are collected in amber glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ±2°C. According to guidelines listed in SW-846 for the extraction of semivolatile compounds, water samples must be extracted within 7 days of collection, and soil samples must be extracted within 14 days of collection. All extracts must be analyzed within 40 days of sample preparation.

1.3.2 Although not a routine sampling practice, a soil holding time study performed by the U.S. Army Cold Regions Research and Engineering Laboratory recommends a maximum holding time of eight weeks when samples are frozen.

1.3.3 A similar study performed for water samples recommends a maximum holding time of 50 days for relatively sterile samples which are refrigerated. Surface waters, or samples likely to have significant microbial activity, may suffer significant losses of nitroaromatic compounds (particularly trinitrobenzene and trinitrotoluene) within 1-2 days, even under refrigeration. If microbial activity is suspected, water samples should be extracted as soon after collection as is practical.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Method Blank Spike	A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

CCV	Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard injected at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.
CF	Calibration factor. The ratio of peak response to nanograms injected. This term is defined in the same way in both the CLP contract and SW-846.
DIW	Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon polishing filters, they are capable of providing organic-free water for use in method blanks and method blank spikes.
IBLK	An instrument blank is solvent containing the method surrogates and is injected into the instrument to monitor for carryover between sample extract injections.
ICV	Initial calibration verification. It is a standard which is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration.
IDL	Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.
MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined at Laucks.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW-846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.

QC Period	Quality control period. An analysis sequence initiated by the injection of one or more standards, followed by sample extracts, and terminated with a standard injection. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
Reporting Limit	Also known as PQL, or Practical Quantitation Limit. This is determined the by low level standard concentration.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the LC column.
RT Window	Retention time window. The ± value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Chromatographic System

Primary Column:	C18 (octadecyl) reverse phase HPLC column, 15 cm x 4.6 mm, 5 µm particle size, (Rainin Microsorb or equivalent).
Secondary Column:	CN (cyano) reverse phase HPLC column, 25 cm x 4.6 mm, 5 µm particle size, (Supelcosil LC-CN or equivalent).
	C8 and CN reverse phase HPLC columns in series, 10cm x 3.9mm, 4µm particle size, (Waters Nova- Pak or equivalent). These are used for the analysis of the 6 additional attenuation compounds.

Mobile Phase:	Methanol (EM Science brand high purity solvent or equivalent). Reagent water (Modulab Polisher HPLC grade water or equivalent).
UV Detector:	254 nm (210 nm for NG and PETN)
HPLC System:	Rainin HPLC system - HPXL solvent delivery system capable of achieving 4000 psi. 50 µL sample loop. Knauer variable wavelength UV detector. Dynamax automatic sample injector. Digital integrator: EZChrom.
Waters System:	Waters 712 WISP Sample Processor or equivalent. 50 µL sample loop or equivalent. Waters 486 tunable absorbance detector or equivalent. Waters 600E Multisolvent delivery system or equivalent. Digital Integrator: EZChrom.
Hewlett Packard System:	HP 1313A Autosampler. HP G1311A QuatPump. HP G1322A On-line degasser. HP G1316A Thermostat column compartment. HP 1314A Variable wavelength detector. Digital Integrator: EZChrom.

2.1.1 Column Temperature Control:

2.1.2 Column temperature is controlled through the use of a column heater which is maintained at a temperature of 25°C.

2.2 Standards

2.2.1 Target Analyte Stock Solution

2.2.1.1 The stock solution used contains the 14 standard compounds in the mix at a concentration of 1000 µg/mL and is generally purchased from AccuStandard Inc. (25 Science Park, New Haven CT 06511). Equivalent solutions from this or other vendors are also acceptable.

2.2.1.2 The NG and PETN stock solutions are purchased individually from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The NG solution comes in 1 mL ampules at 4000

µg/mL in ethanol. The PETN solution comes in 1 mL ampules at 1000 µg/mL in methanol. Equivalent solutions from this or other vendors are also acceptable.

2.2.1.3 The 3,5-Dinitroaniline and 1,3,5-Trinitroso-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from the U.S. Army Environmental Center (Aberdeen Proving Ground, MD 21010). The 2,4-Diamino-6-nitrotoluene, 2,6-Diamino-4-nitrotoluene and 2,2',6,6'-Tetranitro-4,4'-azoxytoluene standards are made from standard analytical reference materials obtained from AccuStandard, Inc. (25 Science Park, New Haven, CT 06511). The 1-Nitroso-3,5-dinitro-1,3,5-hexahydrotriazine standards are made from standard analytical reference materials obtained from Stanford Research.

2.2.1.4 Calibration standard, surrogate stock and IBLK surrogate solutions are detailed in Appendix I.

2.2.2 Surrogate Stock Solution

2.2.2.1 The surrogate stock solution is received in a methanolic solution at a concentration of 1000 µg/mL. The surrogate can also be prepared by weighing 100 mg of 1,2-dinitrobenzene into a 100 mL volumetric flask and diluting to volume with methanol to yield a concentration of 1000 µg/mL.

2.2.3 IBLK Working Solution

2.2.3.1 The working instrument blank solution contains the surrogate compound only at a concentration of 2.0 µg/mL. Prepare this solution by adding 100 µL of the 1000 µg/mL surrogate stock solution to a 50 mL volumetric flask. Dilute to volume so that the final solvent concentration is methanol/water at a ratio of 1:1.

2.2.4 Working Calibration Standards

2.2.4.1 Prepare the working calibration standards for the 14 standard analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis. These 14 analytes are combined into one working solution, since they do not co-elute on the primary column.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentration (µg /mL)
Standard #6	A	0.2	10	20.0
	B	0.2		
Standard #5	C	2.5	10	5.0
Standard #4	C	0.1	2	1.0
Standard #3	C	0.05	2	0.5
Standard #2	C	0.01	2	0.1
Standard #1	C	0.005	2	0.05

Source Solution	Concentration (µg /mL)
A = 14 Component Mix	1000
B = Surrogate Stock Solution	1000
C = Standard #6	20.0

2.2.4.2 Prepare the working calibration standards for the 6 additional analytes in the following manner. All solutions are prepared in acetonitrile and diluted 1:1 with water just prior to analysis.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations (µg /mL)
Standard #6	A	2.0	10	20.0
	B	0.2		
	C	0.2		
	D	0.2		
	E	0.2		
	F	2.0		
Standard #5	H	2.5	10	5.0
Standard #4	H	0.5	10	1.0
Standard #3	I	1.0	10	0.5
Standard #2	J	1.0	10	0.1
Standard #1	K	1.0	10	0.05

Source Solution	Concentration (µg/mL)
A = MNX	100
B = TNX	1000
C = 3,5-DNA	1000
D = 2,6-DA-4-NT	1000
E = 2,4-DA-6-NT	1000
F = 4,4'-TN-AZOXY	100
H = Standard 6	20.0
I = Standard 5	5.0
J = Standard 4	1.0
K = Standard 3	0.5

2.2.4.3 Prepare the working calibration standards for PETN/NG in the following manner. All solutions are prepared in a 1:1 acetonitrile/water mixture.

Standard	Source Solution	Amount Added (mL)	Final Volume (mL)	Final Concentrations NG:PETN:1,2-DNB (µg /mL)
Standard #5	A	0.20	10	20.0
	B	0.10	10	10.0
	C	0.10	10	10.0
Standard #4	D	0.25	0.5	10.0, 5.0, 5.0
Standard #3	D	0.125	0.5	5.0, 2.5, 2.5
Standard #2	D	0.025	0.5	1.0, 0.5, 0.5
Standard #1	D	0.025	1.0	0.5, 0.25, 0.25

Source Solution	Concentration (µg/mL)
A = Nitroglycerin	1000
B = PETN Solution	1000
C = Surrogate Stock Solution	1000
D = Standard 5	As Above

2.2.4.4 Note: Store all standard and surrogate solutions in amber glass vials with screw caps and Teflon-lined septa. Minimize headspace in these vials and store them in a refrigerator kept at 4°C ±2°C. Allow standards to come to room temperature prior to use.

2.2.4.5 Stock solutions may be used for up to one year, and working solutions are good for at least six months, or the expiration date of the parent standard, whichever is sooner.

2.2.4.6 All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP on the traceability, documentation, and preparation standards.

3. Safety precautions

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they are hazardous substances.

3.1.2 All compounds analyzed by this method are used either in the manufacture of explosives or are the degradation products of these compounds. When making stock solutions for calibration, treat each explosive compound with caution.

3.1.3 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.4 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.5 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6 Almost all of the analytes under consideration are known or suspected carcinogens. Analysts should wash their hands after using any standard, solvent or sample extract. Additionally, a respirator should be worn or a fume hood utilized for extremely hazardous compounds or very dirty extracts.

3.2 Waste disposal

3.2.1 Out of date standards and sample extracts are disposed of in the designated organic waste container located in the organic preparation area.

3.2.2 HPLC liquid waste is disposed of by pouring into the designated organic waste container located in the solvent storage area.

4. Operation procedures

4.1 Analytical Conditions

4.1.1 Current C18 Column Conditions for the 14 Compounds:

HPLC3		HPLC4	
Pump A Solvent:	Methanol	Solvent:	Methanol/Water
Pump B Solvent:	Water	Flow:	0-31 min = 0.75 mL/min
Flow:	0.65 mL/minute:		31-48 min = 1.80 mL/min
Gradient:	Linear		48-50 min = 0.75 mL/min
Run Time:	30 minutes	Run Time:	50 minutes
Acquisition Time:	30 minutes	Acq. Time:	50 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

4.1.2 Current CN/Column Conditions for the 14 Compounds:

HPLC4 File #12:

Pump B Solvent:	Water/0.05% H_3PO_4 buffer
Pump C Solvent:	Acetonitrile
Pump D Solvent:	Methanol
Flow:	0.75 mL/minute
Gradient:	Linear
Run Time:	50 minutes
Acquisition Time:	50 minutes

Initial %B	80
Initial %C	10
Initial %D	10

Ramp Time 1:	17 minutes	31 - 48 minutes
Ramp Time 2:	2 minutes	48 - 50 minutes

4.1.3 Current C18 Column Conditions for the 6 Additional Compounds:

Gradient Profile (HPLC3)		**Run Time Profile**
HPLC3 File:	EXTRAS	
Pump A Solvent:	Water *	
Pump B Solvent:	Methanol	
Flow:	1.0 mL/minute	
Gradient:	Linear	
Run Time:	53 minutes	
Acquisition Time:	30 minutes	
Initial %A	90	
Initial %B	10	
Gradient 1 %A:	70	
Gradient 1 %B:	30	
Ramp Time 1:	5 minutes	0 - 5 minutes
Hold Time 1 :	0 minutes	
Gradient 2 %A:	50	
Gradient 2 %B:	50	
Ramp Time 2:	3 minutes	5 - 8 minutes
Hold Time 2 :	7 minutes	8 - 15 minutes
Gradient 3 %A:	26	
Gradient 3 %B:	74	
Ramp Time 3:	2 minutes	15 - 17 minutes
Hold Time 3:	13 minutes	17 - 30 minutes
Gradient 4 %A:	90	
Gradient 4 %B:	10	
Ramp Time 4:	2 minutes	30 - 32 minutes
Hold Time 4:	21 minutes	32 - 53 minutes (Column Equilibration)

4.1.4 Current CN/C8 Column Conditions for the 6 Additional Compounds:

Time (minutes)	Flow (mL/min)	% Methanol	% Water
0	1.0	15	85
2.4	1.0	15	85
7.0	1.0	40	60
10.0	1.0	50	50
17.0	1.0	50	50
20.0	1.0	80	20
21.0	0	15	85

4.1.5 Current Column Conditions for NG/PETN:

C18		CN	
Pump A Solvent:	Methanol	Pump A Solvent:	Methanol
Pump B Solvent:	Water	Pump B Solvent	Water
Flow:	0.7 mL/minute:	Flow: 0.7 mL/min	1.2 mL/minute
Gradient:	Linear (1:1)	Gradient:	Linear (1:1)
Run Time:	30 minutes	Run Time:	15 minutes
Acquisition Time:	15 minutes	Acq. Time:	15 minutes
Injection Volume:	50 µL inj.	Time:	50 µL

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on determination of detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies are performed on both columns. At the time this SOP was written the MDL for the 14 Compounds were between 0.057 mg/kg and 0.64 mg/kg for soils and between 0.071 µg/L and 0.82 µg/L for water. The MDL for the 6 Additional Compounds were between 0.19 mg/Kg and 0.26 mg/kg for soils and between 0.14 µg/L and 0.48 µg/L for water. The MDL for NG/PETN were 0.58 mg/kg and 0.31 mg/kg for soils and 0.16 µg/L and 0.18 µg/L for water respectively.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.4 Retention Time Windows

4.4.1 It is necessary to establish retention time windows for the method by analyzing standards for all target analytes over at least a 24-hour period. A 24-hour time period may be deemed more appropriate for this analysis because the instrument is not always operated for longer periods when analyzing sample extracts. At least 5 standards must be injected during the 24-hour time period, at about equal time intervals. These standards should be interspersed with real sample extracts in order to mimic actual instrument operating conditions. Tabulate the retention times for all standard compounds and compute the sample (n-1) standard deviations of all the retention times.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made or whenever the chromatographic method is modified.

4.4.3 In some cases, the retention time window may be modified in order to take into account any pattern shifting. This shift is acknowledged by observation of the surrogate peak behavior and the surrounding CCV standards. If retention time shift has occurred and the possibility of misidentifying peaks exists, then the sample is reanalyzed bracketed by in-control CCV standards.

4.4.4 See the Laucks SOP on determination of retention time windows for more specifics on their determination and use.

4.5 Initial Multi-Point Calibration

4.5.1 Analyze standard solutions (including the surrogate solution) using at least 5 different concentration levels. The lowest concentration defines the reporting limit. The highest concentration should define the upper usable working range of the detector. Inject the standard solutions from the lowest concentration to the highest. Criteria for evaluating these standards are detailed in Section 6.

4.6 Initial Calibration Verification

4.6.1 At the beginning of an analysis sequence, analyze the mid-point calibration standard (STD 3). The computed calibration factor (CF) or concentration measurement must meet the criteria detailed in Section 6.

4.6.2 Since the retention time windows which were established by the retention time study are relative and not absolute, the windows are anchored by the ICV. This allows the retention times from the ICV to become the mid-point of the retention time windows.

4.7 Instrument Blank

4.7.1 After the analysis of Standard 5, an instrument blank (IBLK) is analyzed. This is to verify that there is no carryover between sample injections. Evaluation criteria are detailed in Section 6.

4.7.2 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK. This IBLK analysis is used only to make a judgment as to the possibility of carryover into the sample analysis immediately following the IBLK. Evaluation criteria are detailed in Section 6.

4.8 Continuing Calibration Verification

4.8.1 A mid-range calibration standard (STD 3) is analyzed after every ten sample injections. In addition, this standard must be the last injection made in the analysis sequence. Evaluation criteria are detailed in Section 6.

4.9 Sample Analysis

4.9.1 Analysis sequence

See Appendix II for a detailed analysis injection sequence.

4.9.2 Sample Preperation

4.9.2.1 Prior to injection on the instrument water sample extracts are diluted 1:1 with water. Any extract that exceeds the instrument calibration range is diluted with 1:1 acetonitrile and water with a dilution factor necessary to quantitate the analytes within the instruments calibration range.

4.9.3 Compound Identification

4.9.3.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the primary column. To confirm the presence of that compound in the sample extract, the peak must also elute in its retention time window on a second column. Currently, separation of the ordnance compounds is achievable. However, when there are analyte co-elutions on the secondary column, the responses are no longer summed. Instead, separate individual standards are analyzed and the sample results are still flagged appropriately. All analyte quantitations come from the C18 column. The CN/C8 columns in

series are typically not used for quantitation purposes. Retention time windows are established as previously described and are updated each QC period. Compounds can only be identified if the ICV and CCV criteria detailed in Section VI are strictly adhered to. Due to constraints of the software, the RT of the co-eluting compounds is determined by assigning the RT to the highest point within a give window. For the co-eluting compounds that may have a slight but not complete separation, such as: 2-nitrotoluene, 3-nitrotoluene and 4-nitrotoluene, this determination of RT can result in RT windows which exceed those identified by the standard (due to the changes in peak heights with concentration values). In order to avoid potential exceedance of RT windows, and in order to accurately identify these co-eluting target analytes, the retention time windows may be administratively set for the co-eluting compounds.

4.9.3.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes. If the concentration of any target analyte exceeds the calibration range, the sample extract must be diluted and reanalyzed.

4.9.4 Compound Quantification

4.9.4.1 Target compound concentrations are calculated using the following equations:

4.9.4.2 Aqueous samples

The external standard equation, as expressed in SW-846 is

$$Concentration(\mu g / L) = \frac{A_x \times A \times V_t \times D}{A_s \times V_i \times V_s}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
A = Amount of standard injected (in nanograms).
A_s = Response for the external standard, same units as A_x.
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if an extract is diluted 10 µL to 1000 µL and subsequently diluted an additional 10 µL to 1000 µL, the expression is: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t = Volume of total extract, µL.

V_s = Initial sample size, mL.

4.9.4.3 In routine use at Laucks, the equation reduces as follows.

4.9.4.4 First, CF is used directly in the equation. Since Cf = A_s/A, this substitution is made. Next, since Laucks routinely measures all final extract volumes in mL, a conversion factor for µL to mL must be made in the numerator of the expression - i.e., V_t = 1000 * mL. Finally, the sample preparation process is represented as the algebraic ratio of initial sample size to final extract volume.

4.9.4.5 The equation then becomes

$$Concentration(\mu g / L) = \frac{1000 \times Ax \times D}{CF \times Vi \times (Vs / Vt)}$$

4.9.4.6 This expression is completely equivalent to the SW-846 equation, yielding the same final result. To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/L * 0.001

4.9.4.7 Non-aqueous samples

4.9.4.8 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Concentration(\mu g / kg) = \frac{1000 \times A_x \times D}{CF \times V_i \times (W / V_t) \times T_s}$$

where:

W_s = Sample size extracted in grams.
T_s = Total Solids in decimal format (i.e. 0.76 not 76).

5. Reports

5.1 Data Packet Organization

See Appendix III for a check list detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike report, and an MS/MSD report.

5.3 Data Qualifying Flags/

5.3.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

U:	The analyte of interest was not detected, to the limit of detection indicated.
B:	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J:	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D:	The value reported is derived from the analysis of a diluted sample or sample extract.
P:	When a dual column technique is employed, this flag indicates that calculated results from the two columns differ by more than 25 percent.
E:	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
X:	The sample has been analyzed at several dilutions. The value reported has been determined to be the most appropriate quantitative value.
Z:	The value reported is from the C18 column and there is coelution with another target analyte on the CN or CN/C8 columns in series.

6. Quality Control

See the Requirements and Corrective Actions table in Appendix IV for additional information.

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 The initial calibration is evaluated by calculating the %RSD of the calibration factors from the five linearity standards.

6.1.1.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

6.1.1.3 This %RSD method assumes a linear response with the calibration curve passing through the origin.

6.1.1.4 The calculated CFs are tabulated and the %RSDs calculated. All compounds must have a %RSD less then or equal to 20% to meet the method criteria.

6.1.2 Corrective action

6.1.2.1 If the %RSD criteria of less then or equal to 20% are not met, the out of control standard should be reanalyzed. If the curve is still out of control, determine the cause of failure and correct. Recalibrate the instrument and reanalyze any samples associated with the out of control curve.

6.2 Initial Calibration Verification

6.2.1 Criteria

6.2.1.1 Using the average CF calculation technique, compute the CFs for each compound. The calibration factors for the ICV standard are compared to the mean CFs for the initial multi-point calibration. The percent difference for these calibration factors is calculated as follows:

$$\%\,Difference = \frac{CF_a - CF_i}{CF_a} \times 100$$

where:
CF_a = .Average CF from the initial multi-point calibration
CF_i = Calibration Factor of the calibration verification standard.

6.2.1.2 The %D cannot exceed 15% for any target analytes or surrogates.

6.2.1.3 The mid-point for the RT window of each compound is updated using the ICV RTs.

6.2.2 Corrective action

6.2.2.1 If the ICV criteria are not met, no sample extracts can be analyzed. Determine the cause of the ICV failure and correct. Reanalyze the ICV and if it is still out of control, a new calibration curve must be analyzed.

6.3 Continuing Calibration Verification

6.3.1 Criteria

6.3.1.1 A CCV standard is analyzed singly for every 10 injections, and after the last sample of the sequence. The CF for each compound is calculated and the percent difference is calculated as shown above.

6.3.1.2 The %D results cannot exceed 15% for any target analytes or surrogates.

6.3.1.3 The retention times for all target analytes must fall within the RT windows established by the ICV.

6.3.2 Corrective action

6.3.2.1 Determine the cause of failure and correct. Reanalyze the calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit in the associated samples.

6.4 Instrument Blank

6.4.1 Criteria

6.4.1.1 There must be no target analyte levels above half the reporting limit in the initial IBLK.

6.4.2 Corrective action

6.4.2.1 If the initial IBLK contains measurable levels of target analytes, the system is out of control. The source of contamination must be identified and corrected.

6.4.2.2 IBLKs are used to monitor for possible carryover in high concentration extracts. Those IBLKs optionally placed into the sequence following suspected high concentration extracts are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be reanalyzed if there is a detectable amount of the analyte found in the IBLK.

6.5 Method Blank

6.5.1 Criteria

6.5.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or at least one blank for every 20 samples, whichever is more frequent. Analytes of interest should not be detected at levels greater than half the current reporting limit, or less than 5% the regulatory limit, or less than 5% of the sample result for the same analyte. If any analytes are detected above half the reporting limit, corrective action must be taken.

6.5.2 Corrective action

6.5.2.1 Steps shall be taken to find/reduce/eliminate the source of the contamination. Sample corrective action may be limited to qualification for blank contamination. When the concentrations of any target analytes in the method blank are above 1/2 the reporting limit for the majority of target analytes or above the reporting limit for target analytes known to be common laboratory contaminants, the effect this may have had on the samples will be assessed. If any analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps shall be taken to find/reduce/eliminate the source in the method blank. The case narrative should aso discuss the situation. If an analyte is found in the method blank and some, or all, of the other batch samples, additional corrective action is required to reanalyze the method blank and any samples containing the same contaminant. If the contamination remains, the contaminated samples of the batch would be reprepared and

reanalyzed with a new method blank and batch specific QC samples. Sporadic cases of contamination may be difficult to control, however, daily contamination would not be acceptable.

6.6 Blank Spike or QC Check Sample (LCS)

6.6.1 Criteria

6.6.1.1 A blank spike follows the same protocol as the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. In addition, this spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. A method blank with added analytes is a blank spike. At the time this SOP was written the blank spike percent recovery control limits were between 30 and 150. These control limits are updated periodically.

6.6.1.2 These control limits are updated periodically.

6.6.2 Corrective action

6.6.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re-extraction and reanalysis would be triggered by any analytes falling outside of control limits in the blank spike sample unless all sample surrogate recoveries and MS/MSD spike recoveries are in control.

6.7 Matrix Spike

6.7.1 Criteria

6.7.1.1 A sample is chosen at random (unless designated by project-specific requirements) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. However, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This frequency may be changed on a project specific basis. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. This spiking solution is supplied from a source other than the calibration standards. The use of a second source for the spiking standards is employed in order to verify the calibration standards. The recovery of spike analytes is calculated as follows:

$$\%Recovery = \frac{SSR - SR}{SA} \times 100$$

where:

SSR = Concentration in spiked sample.
SR = Native concentration in unspiked sample.
SA = Concentration of spike added.

6.7.1.2 At the time this SOP was written the matrix spike percent recovery control limits were between 30 and 150. These control limits are updated periodically.

6.7.2 Corrective action

6.7.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.8 Matrix Spike Duplicate

6.8.1 Criteria

6.8.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2)/2} \times 100$$

where:
S1 = Measured concentration for MS sample.
S2 = Measured concentration for MSD sample.

6.8.1.2 At the time this SOP was written the matrix spike duplicate percent recovery control limits were between 30 and 150. These control limits are updated periodically.

6.8.2 Corrective action

6.8.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. At the time this SOP was written the RPD control limits was 50 percent. These control limits are updated periodically.

6.9 Surrogate Recovery

6.9.1 Criteria

6.9.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\%Recovery = \frac{S_m}{S_a} \times 100$$

where:
S_m = Concentration of surrogate measured in sample.
S_a = Concentration of surrogate added.

6.9.1.2 At the time this SOP was written the surrogate recovery control limits were between 50 and 150 percent. These control limits are updated periodically.

6.9.1.3

6.9.2 Corrective Action

6.9.2.1 Reanalyze the sample. Low surrogate recoveries are greater potential indicators of poor method performance than high surrogate recoveries since non-GC/MS methods cannot separate co-eluting interferents. In instances where high surrogate recoveries are attributable to matrix effects, no corrective action is taken. However, if elevated surrogate recoveries are attributable to preparation error, re-extraction and reanalysis is performed.

6.9.2.2 Check calculations for possible error. Re-extract the sample if surrogate recovery is less than the lower control limit. If a poor injection is suspected, reanalyze the sample.

6.9.2.3 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and reanalyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

7. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Method 8000B, "Gas Chromatography," Revision 1, July 1992, U.S.E.P.A.

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 4th Update, Method 8330A, "Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)," Revision 1, January 1998, U.S.E.P.A.

40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984, U.S.E.P.A.

Special Report 93-11, "Experimental Assessment of Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Soil," Clarence L. Grant et al, June 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

Special Report 93-24, "Evaluation of Pre-extraction Analytical Holding Times for Nitroaromatic and Nitroamine Explosives in Water," Clarence L. Grant et al, August 1993, U.S. Army Cold Regions Research and Engineering Laboratory.

APPENDIX I

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5	STD6
HMX	0.05	0.1	0.5	1.0	5.0	20.0
RDX	0.05	0.1	0.5	1.0	5.0	20.0
1,3,5-TNB	0.05	0.1	0.5	1.0	5.0	20.0
1,3-DNB	0.05	0.1	0.5	1.0	5.0	20.0
Tetryl	0.05	0.1	0.5	1.0	5.0	20.0
NB	0.05	0.1	0.5	1.0	5.0	20.0
2,4,6-TNT	0.05	0.1	0.5	1.0	5.0	20.0
4-Am-DNT	0.05	0.1	0.5	1.0	5.0	20.0
2-Am-DNT	0.05	0.1	0.5	1.0	5.0	20.0
2,4-DNT	0.05	0.1	0.5	1.0	5.0	20.0
2,6-DNT	0.05	0.1	0.5	1.0	5.0	20.0
2-NT	0.05	0.1	0.5	1.0	5.0	20.0
3-NT	0.05	0.1	0.5	1.0	5.0	20.0
4-NT	0.05	0.1	0.5	1.0	5.0	20.0
Surrogate						
1,2-Dinitrobenzene	0.05	0.1	0.5	1.0	5.0	20.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5	STD6
TNX	0.05	0.1	0.5	1.0	5.0	20.0
2,6-DA-4-NT	0.05	0.1	0.5	1.0	5.0	20.0
2,4-DA-4-NT	0.05	0.1	0.5	1.0	5.0	20.0
MNX	0.05	0.1	0.5	1.0	5.0	20.0
3,5-DNA	0.05	0.1	0.5	1.0	5.0	20.0
4,4'-TN-AZOXY	0.05	0.1	0.5	1.0	5.0	20.0
Surrogate						
1,2-Dinitrobenzene	0.05	0.1	0.5	1.0	5.0	20.0

Calibration Standard Solution Concentrations, µg/mL

Compound	STD1	STD2	STD3	STD4	STD5
NG	0.5	1.0	5.0	10.0	20.0
PETN	0.25	0.5	2.5	5.0	10.0
Surrogate					
1,2-Dinitrobenzene	0.25	0.5	2.5	5.0	10.0

Surrogate Stock Solution

Compound	Concentration
1,2-Dinitrobenzene	1000 µg/mL

IBLK Solution

Compound	Concentration
1,2-Dinitrobenzene	2.0 µg/mL

APPENDIX II

Analysis Sequence

Injection	Sample
1	Standard #1
2	Standard #2
3	Standard #3
4	Standard #4(ICV Standard)
5	Standard #5
6	Standard #6
7	IBLK
8	ICV
	injections (9-16)
17	IBLK
18	CCV Standard
	injections (19-27)
28	IBLK
29	CCV Standard
	injections (total of 10 injections between CCVs)
last	IBLK
last	CCV Standard

APPENDIX III

Data Packet Order List

I. QC SUMMARY

Surrogate Recovery Summary Report
Blank Spike Report
MS/MSD Report
Method Blank Summary

II. SAMPLE DATA:

Organic Analysis Data Sheet
Sample Confirmation Worksheet
Chromatogram, primary column
Quantitation Report, primary column
Chromatogram, secondary column
Quantitation Report, secondary column

III. STANDARD DATA:

Linearity Report
ICAL Data
ICAL Response, ICAL std concentrations
ICV reports
CCV reports
Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

Method Blank
Chromatograms
Quantitation Report

Blank Spike
Chromatograms,
Quantitation Report

Matrix Spike
Chromatograms
Quantitation Report

Matrix Spike Duplicate
Chromatograms
Quantitation Report

V. Bench Sheets

SDG Report
Extraction Bench Sheets
Injection Sequence (logbook copy)
Miscellaneous Work Sheets. (%TS, calcs, HTVRs)
Standards Logs

VI. Reject Data:

Data not used to support reported sample results.
All data acquired but rejected on account of QC out of control.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

APPENDIX IV

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Initial Calibration	Minimum of 5 levels.	Minimum of 5 levels. Must demonstrate an RSD of <20%.	As necessary due to major instrument maintenance or difficulties meeting the CCV requirements.	Reanalyze out of control standard. If still out, determine cause of curve failure and correct. Re-analyze curve and any samples analyzed against curve.	Target forms and raw data.
Initial Calibration Verification	+/- 15% of the initial calibration response factor.	+/- 15% of the initial calibration response factor.	Every 24 hr. or at the beginning of an analytical sequence, which ever is more frequent, or as necessary.	Determine cause of ICV failure and correct. Reanalyze ICV and if out of control a new calibration curve must be analyzed.	Target form and raw data.
Continuing Calibration Verification	+/- 15% of the initial calibration response factor. Analyze singly every 10 injections and after the final sample.	+/- 15% of the initial calibration response factor.	Every 10 injections and at the end of an analytical sequence.	Determine cause of failure and correct. Reanalyze calibration curve. All samples bracketed by an out of control CCV must be reanalyzed unless the CCV demonstrates an increase in response and no analytes are detected above the reporting limit.	Target form and raw data.
Instrument Blank	Analyze after analysis of a sample with analyte levels which exceed the upper calibration level.	Analyze instrument blank after samples when high levels of matrix are suspected. Analyze instrument blank before the CCV standard. No target analytes present in the blank > the RL. Surrogate must be within control limit.	Analyze instrument blank after samples when high levels of matrix are suspected. Analyze instrument blank before the CCV standard.	Reanalyze any samples with suspected carryover.	Raw data
Method Blank	All analytes must be < MDL	All analytes must be less than half the Reporting Limit, or <5% regulatory limit, or <5% of the sample result, whichever is greater.	One method blank per 20 samples or each extraction batch, whichever is more frequent.	Reanalyze blank. If still out of control, re-extract the entire batch of samples unless the detected analyte(s) are not present in the associated samples.	Method Blank Summary and raw data. Narrative comment when necessary.

Method 8330 Requirements and Corrective Actions					
QA Element	**Method Criterion**	**Laucks Criterion**	**Frequency**	**Corrective Action**	**Documentation**
Surrogate Recovery	Minimum of 1 surrogate. Recoveries must fall within the labs established windows.	See latest version of Laucks Testing Labs control limits database. All surrogate recoveries must fall within the generated limits. If laboratory limits are not established due to the implementation of a new surrogate , the default limits of 70% - 130% are used until enough data points are collected to generate new limits.	All samples, method blanks, and QC samples.	Re-extract if surrogate recovery is < the lower control limit. If a poor injection is suspected reanalyze the sample.	Target Surrogate Summary form or special test results. Narrative comment when necessary.
MS/MSD	One per 20 samples or extraction batch which ever is more frequent. Must be per matrix.	See latest version of Laucks control limits located in the QC database.	1 MS/MSD pair per 20 samples or every extraction batch which ever is more frequent.	Per SW 846, if analyte recoveries are out of control in the MS/MSD but are in control in the associated blank spike, no further action is required.	MS/MSD report and raw data. Narrative comment when necessary.
Blank Spike	N/A	See latest version of Laucks Testing Labs control limits in the QC database. All recoveries must fall within the generated limits.	1 per 20 samples or every extraction batch which ever is more frequent.	Re-extract if analyte recoveries are outside of control limits.	Blank Spike report and raw data. Narrative comment when necessary.

APPENDIX V

Elution Order of Target Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN/C8) Column
HMX	1	15
RDX	2	11
1,3,5-TNB	3	2
1,3-DNB	5	3
Tetryl	6	14
NB	7	1
2,4,6-TNT	8	9
4-Am-DNT*	9	12
2-Am-DNT*	10	13
2,4-DNT	12	7
2,6-DNT	11	8
2-NT*	13	4
3-NT*	15	5
4-NT*	14	6
Surrogate		
1,2-Dinitrobenzene	4	10

Elution Order of 6 Additional Analytes

Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (C8/CN) Column
TNX	3	1
2,6-DA-4-NT	2	2
2,4-DA-6-NT	1	3
MNX	4	4
3,5-DNA	5	5
4,4'-TN-AZOXY	7	6
Surrogate		
1,2-Dinitrobenzene	6	5

Elution Order of PETN/NG

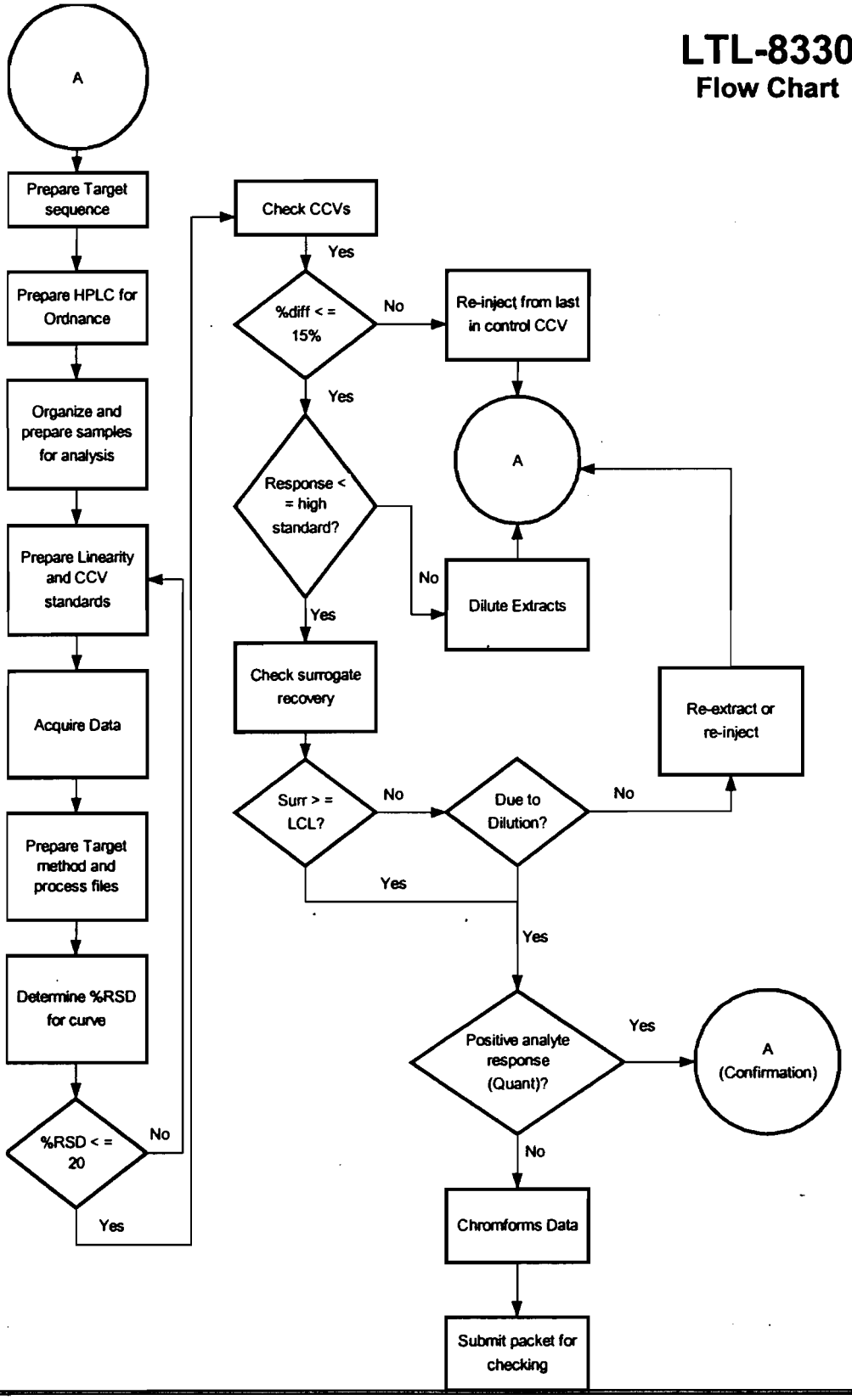
Compound	Order of Elution on Primary (C18) Column	Order of Elution on Confirmation (CN) Column
NG	2	2
PETN	3	3
Surrogate		
1,2-Dinitrobenzene	1	1

*Indicates the compound co-elutes on the confirmation (CN) column.

APPENDIX VI

Method 8330 Flow Chart

LTL-8330
Flow Chart



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP : LTL-9104

Title: **Preparation and Analysis of Total and Amenable Cyanide according to EPA Methods 335.1, 335.3, and SW 846 Method 9012A.**

Revision history:

Number	Date
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2	02/09/98
3	03/07/02

Revised by: _____ Date: 3/11/02
Joan Phillips, Inorganics Supervisor

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Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1 Method Description

1.1.1 Cyanide in samples is determined by a preparation with manual (SW846 Method 9012A) or online distillation (Method 335.3) followed by an automated spectrophotometric analysis on an auto analyzer with a UV digester. The routine reporting limit is 10 µg/L in waters, and 0.50 mg/kg DB in soils and sediments. Pretreatment for the determination of cyanides amenable to chlorination (Method 335.1) in waters is also included in this SOP.

1.1.2 This SOP is applicable to the sample preparation and analysis of cyanide in drinking waters, surface and saline waters, domestic and industrial wastes according to EPA methods 335.1, 335.3 and SW846 Method 9012A with the main difference being the use of the in-line still and UV digestion in EPA 335.3. Also, MIDI distillation with a 50 mL water sample instead of the normal distillation of a 500 mL sample is used in this SOP. Other differences from 9012A are listed on the QA chart at the end of this SOP. Soils and sediments are MIDI distilled as a 1 g sample.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described and in SW846 methodology. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Method Deviations and Comments

1.2.1 The following items represent deviations of Method 9012A, as published, which are followed as standard operating procedures in the performance of this method at Laucks:

1.2.1.1 The MIDI distillation procedure is used, exactly as written in the CLP statement of work. This distillation uses a 50 mL aliquot of sample and a final distillation volume of 50 mls. This procedure allows the lab to use less sample, and dispose of smaller volumes of waste material. All molar ratios in this distillation are identical to the method as written.

1.2.1.2 The calibration curve specified in 9012A is reduced by 2 to compensate for the lack of sample concentration in the distillation step.

1.3 Method Summary

1.3.1 **Sample Preparation:** The MIDI distillation procedure applies to relatively solid samples such as soils, sediments and sludges, or, if requested, to water samples. Cyanide determined by this method is defined as cyanide ion and complex cyanides converted to hydrocyanic acid by reaction in a reflux system with mineral acid in the presence of magnesium ion. The cyanide as hydrocyanic acid (HCN) is released from cyanide complexes by means of a MIDI reflux-

distillation operation and absorbed in a scrubber containing sodium hydroxide solution. The cyanide in the distillate is then determined in a spectrophotometric analysis.

1.3.2 Water samples analyzed under EPA 335.3 are not externally distilled and are directly analyzed unless otherwise requested.

1.3.3 **Spectrophotometric Analysis:** On a Technicon auto analyzer, the samples are acidified and passed through a UV digester and then distilled in-line. The cyanide released is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH less than 8 without hydrolysis to the cyanate. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 570 nm. To obtain colors of comparable intensity, it is essential to have the same salt content in both the samples and the standards.

1.4 Sample Handling, Sample Storage, Holding Times

1.4.1 Samples are preserved with 8 pellets of sodium hydroxide per liter of sample (pH > 12) at the time of collection. Samples must be stored at 4°C ± 2°C and must be analyzed within the specified holding time of 14 days from date of collection. The samples can be submitted in either plastic or glass containers. The pH is checked at the time of sample receipt and at the time of analysis and the client should be notified if the pH is less than 12.

1.4.2 Samples are to be checked for chlorine interference at the time of collection and the appropriate action should be taken.

1.5 Interferences

1.5.1 Sulfide interference: Some sulfides convert CN^- to SCN^-. Add cadmium carbonate to sample until lead acetate test paper no longer darkens (treat 25 mL more than is necessary for the distillation). Yellow cadmium sulfide precipitates if the sample contains sulfide. Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss by complexation or occlusion of cyanide on the precipitated material. Filter sample through a dry filter paper into a dry container then proceed with the distillation procedure. A narration is included in the final report. Run a standard treated in the same manner with the treated samples.

1.5.2 Chlorine interference: Chlorine decomposes most cyanides. Add ascorbic acid to the 50 mL sample ~0.02 g at a time until KI-starch test paper no longer turns bluish then add an additional 0.02 g of ascorbic acid. Proceed with the distillation procedure. A narration is included in the final report.

1.5.3 NO_3 / NO_2 interference: Nitrates and/or nitrites can form oximes with organic compounds which can decompose under test conditions to form HCN. If high levels are suspected (e.g. fertilizers) then sulfamic acid is added to the sample to remove the interference.

1.5.4 Surfactants. For manual distillation, the presence of surfactants, detergents or fatty acids may cause the sample to foam during refluxing. If this occurs, add an antifoam agent such as Baker Antifoam B Silicone Emulsion.

1.5.5 Interferences are eliminated or reduced by the distillation procedure

1.5.6 Thiocyanates are a positive interference. During the UV Digestion thiocyanates are decomposed to cyanide.

1.6 Definition of Terms

1.6.1 This section defines terms and acronyms as they are used in this SOP. Other terms are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Batch Identifier - A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. The batch identifier for the second set of water samples analyzed on Feb 6, 1998 would be A020698AA1W02.

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing Calibration Verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC, r - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run and compared to the initial calibration to determine whether the instrument calibration is accurate.

MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment, Reagents, and Standards

2.1 Equipment

2.1.1 Required instrumentation includes the following:

2.1.1.1 Midi reflux distillation system with heating block capable of maintaining 125°C ± 5°C. We use the MIDI-VAP model MC-100 ("CYN-TEN").

2.1.1.2 Technicon Auto-Analyzer with in-line distillation unit and UV digester.

2.1.1.3 Any laboratory PC equipped with Excel and the Cyanide template may be used for calculation of data.

2.2 Reagents

2.2.1 0.25 N NaOH: dissolve 10.0 g NaOH pellets in 1 L DIW. CAUTION: this reagent will get hot upon mixing!

2.2.2 $MgCl_2$ solution: dissolve 510 g $MgCl_2 \cdot 6H_2O$ and dilute to 1 L with DIW.

2.2.3 1:1 H_2SO_4: carefully and slowly add with mixing 500 mL of conc. H_2SO_4 to 500 mL of DIW. CAUTION: this reagent will get **EXTREMELY** hot upon mixing! Mix in a Pyrex or equivalent container! **DO NOT ADD WATER TO ACID!**

2.2.4 Phosphate buffer: dissolve 13.6 g monobasic potassium phosphate (KH_2PO_4) and 0.28 g anhydrous dibasic sodium phosphate (Na_2HPO_4) and dilute to 1 L with DIW.

2.2.5 Chloramine -T solution: dissolve 1.0 g in 250 mL of DIW. Prepare this reagent fresh daily.

2.2.6 Color reagent: place 15 g of barbituric acid in the bottom of a container, and rinse down the sides of the container with DIW. Add 100 mL of DIW and swirl gently. Add 75 mL of pyridine and mix. Add 15 mL of conc. HCl, mix, and cool. Dilute to 1 L with DIW. This reagent should be stored at 4°C. This reagent should be made under a hood.

2.2.7 Distillation reagent: to 500 mL of DIW add 250 mL conc. H_3PO_4 and 50 mL 50% H_3PO_2 and then dilute to 1 L with DIW.

2.2.8 Sodium Hypochlorite solution: Commercial bleach such as Clorox (5.5%).

2.3 Standards

2.3.1 KCN stock cyanide solution for standards and spiking, 100 mg/L CN. Place 0.2510 g of KCN and approx. 1.5 g NaOH in a 1.0 L volumetric flask and dissolve. Dilute up to the mark with DIW. Record this standard in a standards logbook. The stock standard is good for one year.

2.3.2 Intermediate cyanide standard solution, 1.00 mg/L CN. Dilute 1.0 mL of stock standard solution 2.3.1 to 100 mL with 0.25 N NaOH. Prepare fresh solution at least weekly and record in a standards logbook.

2.3.3 Blank solution for zero standard, blanks and diluting samples and standards: Dilute 200 µL 10 N NaOH to 100 mL with DIW. Prepare fresh solution at least weekly and as needed.

2.3.4 Calibration standards:

2.3.4.1 Method 9012A

Concentration (mg/L)	µLs of 2.3.2	Final volume (mL)
0.200	1000	5.0
0.150	750	5.0
0.100	500.	5.0
0.050	250.	5.0
0.020	100	5.0
0.010	50.	5.0
0	0	5.0

All standards should be diluted up to final volume with 2.3.3 Blank solution. Standards are consumed during analysis so prepare fresh standards for each analytical run.

2.3.4.2 Method 335.3

Concentration (mg/L)	µLs of 2.3.2	Final volume (mL)
0.250	1250	5.0
0.100	500	5.0
0.050	250	5.0
0.010	50	5.0
0	0	5.0

All standards should be diluted up to final volume with 2.3.3 Blank solution. Standards are consumed during analysis so prepare fresh standards for each analytical run.

2.3.5 SRM/ICV stock solution: dissolve 0.2115 mg potassium ferricyanide ($K_3Fe(CN)_6$) and approximately 1.5 g NaOH in DIW and dilute to 1 L in a volumetric flask. This stock standard has a cyanide concentration of 100 mg/L and should be stored at 4°C ± 2°C. This standard expires after one year.

3. Safety Precautions

3.1 Refer to the Laucks SOP on lab safety practices and the instrument manufacturer's manual for routine instrument precautions.

3.2 All standards, samples and sample solutions should be handled as if they are hazardous substances. Some forms of cyanide can be released as a gaseous form upon acidification. Treat all samples as if they definitely contain cyanide.

3.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.4 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.5 Sodium hydroxide and sulfuric and phosphoric acid are extreme irritants if they come in contact with skin or eyes. When using these chemicals, the analyst should wear gloves and safety glasses.

3.6 The color reagent (containing pyridine) should be prepared under a hood.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on determining detection limits. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration 1-5 times the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. The LTL estimated water MDLs are 0.002 mg/L for EPA method 335.3 and SW846 method 9012A and 0.3 mg/Kg for SW846 method 9012A for soils.

4.2 Initial Multi-Point Calibration

4.2.1 The calibration standards listed in section 2.3.4 should be analyzed in order. This calibration MUST IMMEDIATELY PRECEDE sample analysis.

4.2.2 The highest standard used in the linear regression calculation defines the upper usable working range of the instrument. Any sample concentration value that exceeds the concentration of this high standard must be diluted with blank solution and reanalyzed.

4.2.3 Criteria

4.2.3.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The linear correlation coefficient must be 0.995 or greater. All CCVs and sample concentrations must be computed using the regression equation.

4.2.4 Corrective action

4.2.4.1 If the criteria are not met the instrument must be recalibrated. No data may be reported for an analyte that does not have a valid calibration.

4.2.5 Calibration for samples containing sulfide

4.2.5.1 Five standards of similar concentration to those in section 2.3.4 should be distilled in the same manner as samples, adding similar amounts of cadmium carbonate to all standards.

4.3 Initial Calibration Verification

4.3.1 At the start of each analytical run, analyze a standard from a source other than that from which the calibration material was obtained.

4.3.2 For method 335.3, the ICV/SRM is prepared by diluting 0.010 mL of Potassium Ferricyanide stock solution to 10 mL with blank solution.

4.3.3 For SW846 Method 9012A, the ICV/SRM is prepared by spiking 250uL of Potassium Ferricyanide stock solution to 50 mL of DIW and distilling along with the samples. The distillate is diluted by a factor of 10 at the instrument.

4.3.4 This standard must be prepared with every 20 samples or with every batch, whichever is more frequent. Only the first ICV/SRM is considered an ICV for the analysis.

4.3.5 Criteria

4.3.5.1 The calculated concentration of the ICV/SRM must be within the limits supplied by the manufacturer or not to exceed ±15% of the true value.

4.3.6 Corrective action

4.3.6.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated. No data can be reported for an analyte that does not have a valid ICV.

4.4 Initial Calibration Blank

4.4.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed ½ of the reporting limit.

4.4.2 Corrective action

4.4.2.1 If the ICB contains target analyte levels above ½ of the reporting the system is considered to be out of control. The source of contamination must be identified and corrected

before proceeding with the analysis. No data can be reported for an analyte that does not have a valid ICB.

4.5 Blank Spikes

4.5.1 For Method 9012A, standards spiked with 10µL and 50µL of the 100mg/L KCN stock standard are distilled with every batch. These standards are analyzed immediately following the ICB. These standards must agree within ± 10% of the undistilled standard before continuing analysis. If the standards do not meet this criteria, the cause of the error should be found before proceeding.

4.6 Method Detection Limit Standard

4.6.1 After the analysis of the ICV and the ICB, but before the analysis of any sample extracts, an MDL standard is to be analyzed. The MDL standard is used to provide on-going verification of the ability of the system to detect analytes at a concentration near the method detection limit. The MDL standard should read between 67% and 220% of the true value. This limit may be overlooked if the recovery of this check standard is high. It must, however, be detected for the system to be considered in control.

4.6.2 Corrective Action

4.6.2.1 If target analytes are not detected, the analysis must be terminated until the problem has been solved. Alternatively, if the affected samples are well above the detection limit (i.e. bracketed by appropriate standards), they may be reported. No undetected values should be reported if the MDL standard for that analyte(s) is undetected.

4.7 Continuing Calibration Verification (CCV) and Blank (CCB)

4.7.1 A mid-range calibration standard is analyzed at a minimum of once every 10 sample injections. Immediately following the CCV, a blank solution is analyzed. In addition, at the end of the run, this standard should be run at a different level than preceding CCVs to verify that the curve has remained linear throughout the analysis. A CCB should follow this final CCV.

4.7.2 Criteria

4.7.2.1 The CCV must fall within ± 15% of the true value.

4.7.2.2 The levels of target analytes in the CCB should not exceed ½ of the reporting limit.

4.7.3 Corrective action

4.7.3.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a

successful ICV and a CCV which is in control or by preceding and following CCVs which are within limits.

4.7.3.2 If the CCB contains target analyte levels above ½ of the reporting limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank that are in control or by preceding and following CCBs that are within limits.

4.8 Method Blanks

4.8.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time, or at least one blank every 20 samples, whichever is more frequent. Any analyte response above the reporting limit is reported. Method blank control limits are that contamination should not exceed ½ of the reporting limit for waters.

4.8.2 For manual distillation preparation (Method 9012A), the method blank consists of a 50 mL sample of DIW and is used to verify contamination free reagents and apparatus. A Method Blank is distilled every 20 samples or with every batch whichever is more frequent.

4.8.3 For autoanalyzed water samples (Method 335.3), a method blank is analyzed for every 20 samples analyzed. The method blank consists of a sample of blank solution.

4.8.4 Corrective action

4.8.4.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.9 Matrix Spike/Matrix Spike Duplicate

4.9.1 Project-specific criteria will determine whether MS/MSD or MS/Duplicate analysis is to be performed. MS/MSD Duplicate precision is calculated as in Section 4.9 – precision usually is project-specific.

4.9.2 For manual distillation preparation 10 µL of 100 mg/L CN standard solution is added to the 50 mL of sample prior to distillation, for a spike value of 0.100 mg/L in the 50 mL of distillate.

4.9.3 For autoanalyzed water samples the 10 µL of the 100 mg/L CN standard is added to 10 mL of sample prior to analysis for a spike value of 0.100 mg/L.

4.9.4 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS - S) * 100}{SA}$$

where: SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

The recovery criteria are detailed in the current in the Quality Control Database and will change from time to time. The current water control limits are 64-135% recovery; the current soil control limits are 50-150% recovery.

4.9.5 Corrective action

4.9.5.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.10 Sample Duplicate

4.10.1 Criteria

4.10.1.1 Sample duplicates are required when running CLP practices are employed.. At least one duplicate sample per 20 samples per matrix is required. RPD values are calculated in the following manner:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where: S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

The RPD control limits are detailed in the Quality Control Database and will change from time to time. The current water control limits are 11% RPD; The current soil control limits are 30% RPD.

4.10.2 Corrective action

4.10.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Pre-distillation Sample Screen for Waters

5.1.1 Water samples are screened for sulfide and chlorine interferences prior to distillation as follows:

5.1.1.1 Pour a small amount of pH 4 buffer into a 1oz. plastic cup.

5.1.1.2 Check for the presence of sulfide with lead acetate test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A darkening of the paper indicates the presence of S^{2-}. If present then see Section 1.4 Interferences before proceeding with the distillation.

5.1.1.3 Check for the presence of chlorine with potassium iodide-starch test paper. Wet the paper with pH 4 buffer and then drop ~10 µL of sample onto it. A bluish discoloration indicates the presence of chlorine. If present then see Section 1.4 Interferences before proceeding with the distillation.

5.1.2 Any interferences are noted on the bench sheet.

5.2 Pretreatment for Cyanides Amenable to Chlorination

5.2.1 Two sample aliquots are required for chlorine amenable cyanide determination. One is pretreated, the other is not pretreated. Both are prepped and analyzed and the difference between the two is the cyanide amenable to chlorination.

5.2.1.1 To the sample aliquot designated for pretreatment add bleach solution dropwise until an excess of chlorine is maintained. Test for residual chlorine with KI-starch paper. Paper will turn blue.

5.2.1.2 Maintain excess for one hour with continued agitation using a Teflon stir bar and a stir plate.

5.2.1.3 After one hour, add ascorbic acid in small increments (~0.01 g) until KI-starch paper shows no residual chlorine and analyze.

5.3 Manual Distillation

5.3.1 Samples are distilled using the MIDI distillation method. The distillation procedure is as follows:

5.3.1.1 Remove fritted tubes from absorbers, rinse, and set aside.

5.3.1.2 Rinse out absorbers, and invert to drain.

5.3.1.3 Add 50.0 mL of a liquid sample (or 1.0 g of a solid sample) to a sample tube. For solid samples, dilute with DIW to the 50 mL mark on the tube.

5.3.1.4 Set up sample tubes for the appropriate QC (SRM, Method Blank, MS/MSD), but do not spike the SRM or the MS/MSD samples yet.

5.3.1.5 Add 3-4 boiling stones to each tube.

5.3.1.6 Insert reflux impingers into the sample tubes.

5.3.1.7 Fill the absorber tubes with 50.0 mL of 0.25 N NaOH, and insert the fritted tubes.

5.3.1.8 Place sample tubes in the back row of the heating block, and the absorbers into the front row.

5.3.1.9 Insert the cold-finger condensers into the top of the sample tubes.

5.3.1.10 Hook up all vacuum hoses.

5.3.2 Turn on water flow, and adjust flow meter to approx. the same number of samples that are being distilled (about 9-10 for 10 samples).

5.3.2.1 Plug in the vacuum pump.

5.3.2.2 Stabilize the flow rate at about 3 bubbles per second and wait for about 5 minutes.

5.3.2.3 Spike the SRM and the MS/MSD with the appropriate spiking material.

5.3.2.4 Add 5 mL of 1:1 H_2SO_4 to each tube, and let mix for about 3 minutes.

5.3.2.5 Add 2 mL $MgCl_2$ to each tube, and rinse tubes with DIW.

5.3.2.6 Turn power on, and turn knob to mark.

5.3.2.7 When distillation is complete (10 minutes to heat to reflux, 60 mins of reflux, and 15 minutes of cooling) the heating block will automatically shut itself off. Replace tubes in tube rack, removing the fritted tubes before turning the vacuum pump off.

5.3.2.8 Pour off the liquid from the absorbers into a clean container, label, and store at 4°C if the distillates will not be analyzed immediately.

5.3.2.9 Rinse all glassware with DIW, and store fritted tubes in DIW.

6. Sample Analysis Using a Technicon Auto Analyzer

6.1 Instrument start-up and operation

6.1.1 The auto-analyzer should be set up for cyanide analysis according to the Laucks SOP on configuring the Technicon Autoanalyzer. Turn on power and allow heating bath to reach 150 degrees C while flushing reagent tubes with DIW. After the temperature is reached, place the reagent tubes into their proper reagents. Turn on chart drive motor and allow reagents to flow through completely. When baseline has settled, set baseline at about 90%T.

6.2 Analysis procedure

6.2.1 Prepare the 1.0 ppm working standard and the blank solution

6.2.2 From these solutions make the standard curve in 1 oz plastic cups, swirl and transfer to sampler cups, and load onto sampler.

6.2.3 Immediately after the calibration standards, load an EPA approved LCSW as the ICV. Immediately after the ICV, load the blank solution for the ICB.

6.2.4 Load standards, water samples, and distillates from manual distillation (distillates are analyzed the same as water samples) as in the following example and press the reset button on the autosampler to begin the analysis:

Method 9012A

cup#	cup contains
1	STD 1 0 ppb
2	STD 2 10 ppb
3	STD 3 20 ppb
4	STD 4 50 ppb
5	STD 5 100 ppb
6	STD 6 150 ppb
7	STD 7 200 ppb
8	ICV/LCSW1
9	ICB/PBW1
10	Blank spike low
11	Blank spike high
12	Sample 1
13	Sample 1MS
14	Sample 1MSD
15	Next Sample
16	...
17	CCVx
18	CCBx

Method 335.3

cup#	cup contains
1	Std 1 0.250 mg/L
2	Std 2 0.100 mg/L
3	Std 3 0.050 mg/L
4	Std 4 0.010 mg/L
5	Std 5 0 mg/L
6	ICV/LCSW1
7	ICB/PBW1
8	MDL
9	Sample 1
10	Sample 1MS
11	Sample 1MSD
12	Next Sample
13	...
14	CCVx
15	CCBx

6.2.5 After sampling is finished and the last sample peak has been recorded, turn off the chart drive motor, put reagent tubes into wash and turn off the oil bath heater. Once the oil bath has cooled below 100 degrees C, the instrument can be entirely shut down.

6.3 Compound Quantification

6.3.1 The data for each sample is recorded in strip-chart form. On a bench sheet is recorded the information for each sample such as Sample ID, Cup #, and Dilution. Then the value for the Peak Ht and Baseline, as read from the strip-chart (to the nearest 0.25 units), is recorded for each sample. This data is then entered into a standard spreadsheet (Excel or equivalent) for the calculation of all other values. (An example of the spreadsheet is included in appendix II.)

6.3.1.1 Percent transmittance is calculated from the formula:

$$\%T = \text{Peak Ht}/(\text{Baseline}/100)$$

where the ratio Baseline/100 accounts for baseline drift.

6.3.1.2 Absorbance is calculated from %T by the formula:

$$\text{Abs} = 2\text{-log}(\%T)$$

6.3.1.3 The spreadsheet performs a linear regression including the calibration standards 0 mg/L up to 0.200 mg/L with x being the true value of the standards, and y being their corresponding absorbances.

6.3.1.4 Concentration of CN^- in the samples is calculated from the linear regression curve using the formula y=mx+b. Substituting the sample absorbance as the y value and solving for x (conc. of CN^- in mg/L) and multiplying by the dilution factor, the formula becomes:

conc. of CN^- mg/L = [(Abs. - y intercept) / slope] * dilution

6.3.2 For soil/sludge samples, the mg/L integrator value is converted to mg/Kg DB by the following equation

$$\text{Soil:} \quad Cs = \frac{Ci * Vs * 100}{Ws \; TS}$$

Cs = Sample concentration (mg/kg DB)
Ci = Concentration in sample injected (mg/L)
Vs = Volume of extract (mL)
Ws = Weight of sample extracted (gm)
TS = Total solids (represented as decimal)

7. Reports

7.1 Sample Results and Quality Control Reports

7.1.1 Data packages shall consist of the sample results summary, the chromatograms for samples in the workorder, a copy of the calibration curve and standard log book, and a copy of the QC summary report form. These include the method blank analysis, matrix spike results, duplicate results, and the Standard Reference Material (SRM) reports. A copy of the QC summary report form must be included in order to indicate that the QC was evaluated for analytical consistency.

7.2 Data Qualifying Flags

7.2.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.
*	: QC limits were exceeded.

Appendix I
Laucks Testing Laboratories
Method 335.1, 335.3 and SW846 9012A QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	9012 A: 7 standards plus a blank. r ≥0.995 335.3: No criteria	6 stds plus a blank r ≥0.995 (standards must be distilled if sulfide is found in samples) 4 standards plus a blank r ≥0.995	Beginning of run	Recalibrate if r < 0.995	In raw data.
Initial Calibration Verification / LCSW	Verify calibration every 15 samples with an independently prepared check standard.±15%.	Independent source ±15%.	1 per batch or 1 per 20 samples including one immediately after calibration.	Reanalyze, if reanalysis fails recalibrate and or perform instrument maintenance. No data can be reported if ICV fails.	In the raw data package, and in the quality control data base.
Initial Calibration Blank	No criteria.	less than ½ the reporting limit	1 per run immediately after ICV.	Reanalyze. No data can be reported if ICB fails.	In the raw data package.
Continuing Calibration Verification	Verify calibration with the mid-range standard. Must agree within 15% of true value.	Verify calibration with the mid-range standard. Must agree within 15% of true value.	1 after every 10 samples, and at the end of the run.	Samples which are not bracketed by in control CCVs must be reanalyzed.	In the raw data package.
Continuing Calibration Blank	No criteria.	less than ½ the reporting limit	1 after every 10 samples, and at the end of the run.	Samples which are not bracketed by in control CCBs must be reanalyzed.	In the raw data package.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank Soil: Water:	 No criteria. No criteria.	less than ½ the reporting limit	1 per batch or 1 per 20 samples.	If sample concentration exceed 10x the contamination level, no action required. Samples with contaminated Method Blanks must be re-prepared and reanalyzed.	In the raw data package, and in the quality control data base.
Matrix Spike (%Rec) Soil: Water:	Run one spike duplicate every 10 samples. No recovery criteria .	Water 64-135% Soil 50-150%	1 per batch or 1 per 20 samples.	Review data, consult QC officer, some conditions may require reanalysis.	In the raw data package, and in the quality control data base.
Matrix Spike Dup. (RPD) Soil: Water:	See MS.	Water 11% Soil 30%	1 per batch or 1 per 20 samples.	Review data, consult QC officer, some conditions may require reanalysis.	In the raw data package, and in the quality control data base.
Distilled SRM (%Rec) (Manually distilled soils and waters only.)	Independent check sample	+/- 15% of true value.	1 per batch or 1 per 20 samples.	Reprep and reanalyze.	In the raw data package, and in the quality control data base.
Distilled Blank Spikes	9012A: two standards, one low, one high 335.3: No criteria	+/- 10% of undistilled standards	2 standards, one high, one low, per batch.	Reprep and reanalyze.	In the raw data package, and in the quality control data base.

Appendix II

Excel spreadsheet used for cyanide calculations.
(example)

Cyanide Analysis	ITEM	DATE	METHOD	ANALYST	CHECKER	
JOB#:	prep.		Man. Distill	PL		
MATRIX: water	analysis	02/14/00	EPA 335.3	PL		

STANDARD CURVE LCSW: WP586 (tv=0.100 ppm)

standard stock:
TE4-67-17

	T.V. (mg/L CN)	Peak Ht.	Baseline	%T	Absorbance
1	0.250	16.00	92.00	17.39	0.760
2	0.100	42.50	89.00	47.75	0.321
3	0.050	61.00	89.50	68.16	0.166
4	0.010	82.50	90.00	91.67	0.038
5	0.000	89.00	89.50	99.44	0.002
6	--x value--				--y value--

r= 0.9997 m= 3.0173 y(0)= 0.0101 x(0)= -0.003

ms/msd=0.100ppm

	Sample I.D.	Dilution	Peak Ht.	Baseline	%T	Absorbance	CN (mg/L)	
7	ICV / LCSW1	5	41.00	95.00	57.11	0.337	0.1082	108% Rec.
8	ICB	1	87.5	95.00	99.21	0.002	-0.0025	0.005 U
9	MDL tv=0.005 mg/L	1	84.00	95.00	96.05	0.020	0.0034	68% Rec.
0	b021400cnw01	1	86.50	95.00	99.47	0.003	0.0116	0.005 U
1	0005555-2	1	78.00	95.00	99.47	0.045	-0.0025	0.005 U
2	0005555-1	1	85.00	95.00	50.53	0.003	0.1395	140% Rec.
3	0005555-1ms	1	31.50	95.00	50.79	0.431	0.1325	132% Rec.
4	0005555-1msd	1	32.50	95.00	99.21	0.410	0.0004	0.005 U
5		1						
6		1						
7		1						
8		1						
9	CCV1 tv=0.100 mg/L	1	28.50	82.50	47.89	0.243	0.1082	108% Rec.
0	CCB1	1	83.00	83.00	98.95	0.005	0.0008	0.005 U

Appendix III

Sample Bench Sheet

Cyanide Analysis Run Log

Date	Method	Analyst
3/5/02	EPA 335.3	NS

Intermediate Std: TE6-45-4 tv=1.00 mg/L

LCSW: APG 26602 tv=0.630 mg/L

Cup #	Sample ID	Peak Ht.	Baseline	Dilution (mL/mL)	pH	Cl_2	S^{2-}
1	Sys. Rinse	92.00	92.50				
2	0.250 mg/L	16.00	92.00				
3	0.100 mg/L	42.50	89.00				
4	0.050 mg/L	61.00	89.50				
5	0.010 mg/L	82.50	~~89.50~~ 90.00				
6	0.000 mg/L	89.00	~~89.50~~ 89.50				
7	ICV/LCSW1	41.00	89.00				
8	ICB	87.50	88.00				
9	MDL	84.00	86.00				
10	PBW1	86.50	87.00				
11	0203024-1	78.00	86.50		7.12	–	–
12	0203029-1	~~85.50~~ 85.50	85.50				
13	IMS	31.50	85.00				
14	IMSD	32.50	83.50				
15							
16							
18							
19	CCV1	33.00	82.50				
20	CCB1	83.00	83.00				
21							
22							
23							
24							
25							
26							
27							
28							
29							
30							
31	CCV2						
32	CCB2						

Cup #	Sample ID	Peak Ht.	Baseline	Dilution (mL/mL)	pH	C
33	–					
34						
35						
36						
37						
38						
39						
40						
41						
42						
43	CCV3					
44	CCB3					
45						
46						
47						
48						
49						
50						
51						
52						
53						
54						
55	CCV4					
56	CCB4					
57						
58						
59						
60						
61						
62						
63						
64						
65						
66						
67	CCV5					
68	CCB5					

ck for interferences from sulfides and chlorine

P (Pass): test paper did not change color

F (Fail): test paper changed color

Appendix III

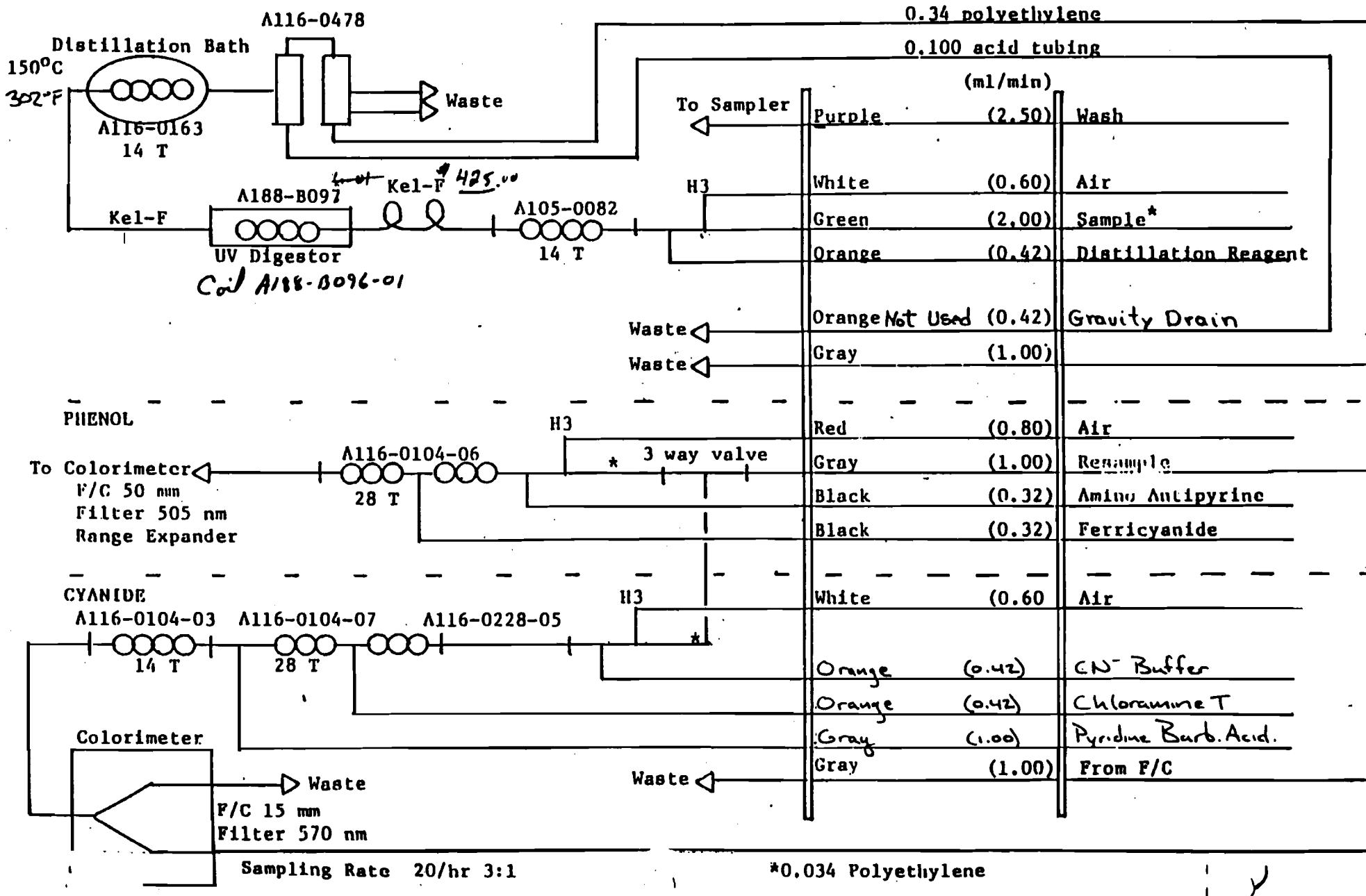
Autoanalyzer Flow Diagram

[illegible] chemistry
currently in use.
Jan 29, 1981 MS

CYANIDE-PHENOL
Combin[illegible] Manifold
AAI

ALPKEM Co[illegible]ation
1981
RRP

DISTILLATION



Sampling Rate 20/hr 3:1

*0.034 Polyethylene

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9110

Determination of Anions by Ion Chromatography by EPA Method 300.0A or SW846 Method 9056

Revision history:

Number	Date
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7	03/01/01
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5	1/12/98
4	3/08/97
3	6/19/96
2	5/08/96
1.1 (LTL-0030)	5/31/90
1.0 (LTL-0030)	10/19/89

Written by: _____ Date: 3/8/02
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Reviewed by: _____ Date: 3-8-02
Harry Romberg, QA Officer

Approved by: _____ Date: 3-11-02
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 **Assigned to:** Tetr

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method describes the technique used to determine anions in water using Ion Chromatography. The method is capable of being extended to the analysis of anions in soil matrices if a preliminary leaching step is performed to solubilize the anions. Also, anion determinations may also be performed on the solution resulting from the oxygen bomb decomposition of fuels, solids, oils, and total/organic halogen extracts. The ions that may be quantified by this method are fluoride, chloride, nitrite, nitrate, sulfate, bromide and phosphate. **However, this is a trace-level method and, in many cases, it may be preferable to use a colorimetric/turbidimetric method.**

1.1.2 The anions in an aqueous solution are introduced via an autosampler into a flowing eluant stream. This stream is passed through a cation exchange column, which strips the metallic elements from the solution. It then passes through an anion exchange column where the chromatographic separation occurs. The separation column effluent is then passed through an anion micromembrane suppresser where counter ions are exchanged for hydronium ions. This serves to suppress the conductance of the eluant and enhances the conductance of the anionic analytes of interest by forming their complementary acids. Following this exchange the stream is passed through a conductivity detector. The detector output is collected by a data acquisition system, which stores the chromatograms for later calculation and interpretation.

1.1.3 The method is based on EPA 300.0 A, or SW-846 9056. Method choice is based on client request or environmental program. The following table summarizes the differences between the two methods.

Method 300.0 Criteria	Method 9056
CCV must be ±10%	CCV must be ±5%
Duplicate frequency is 1/20	Duplicate frequency is 1/10

1.1.4 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are collected in 500 or 1000 mL unpreserved polyethylene bottles. Upon receipt, samples are stored 4° ± 2° C.

1.2.2 Analysis holding time is dependent upon the anion of interest. Holding time for Nitrate, Nitrite, and Ortho phosphate analysis is 48 hrs. Holding time for Bromide, Chloride, Fluoride, and Sulfate is 28 days.

1.3 Interferences

1.3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

1.3.2 The water dip or negative peak that elutes near and can interfere with the fluoride peak. Care should be taken so that integration is being done properly.

1.3.3 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in IC.

1.3.4 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

1.3.5 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L this interference may not be significant. The analytical column AS14 now in use, minimizes this problem.

1.3.6 The acetate anion elutes early during the chromatographic run. The retention times of the anions seem to differ when large amounts of acetate are present. Therefore this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

1.4 Definition of Terms

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline and to estimate carry-over from a mid-level standard (CCV).

CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC, r - Correlation coefficient (r)- A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is within control.

MDL - Method detection limit - The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are determined empirically by Laucks. See section 4.1 for a more detailed discussion of MDL determination.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

RT - Retention time - The time (in minutes) at which a target analyte elutes from the IC (or other) column.

RT window - Retention time window - The +/- value which is applied to the individual analyte peaks to establish the time range used to make tentative target analyte identifications.

Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Ion chromatograph, Dionex model DX100 or equivalent consisting of a single piston pump, autosampler, either manual or automated injector, guard column, separator column, anion micromembrane suppresser, and a conductivity detector.

2.1.2 Guard column, Dionex AG14 or equivalent

2.1.3 Chromatographic column, Dionex AS14 or equivalent

2.1.4 Anion Micro Membrane Suppresser, Dionex ASRS-1 or equivalent

2.1.5 Electronic data collection device, AI-450 Chromotography Software or equivalent, or other applicable data collection system.

2.2 Standards and Solutions

2.2.1 Calibration Standard/Spiking Solution.

2.2.1.1 A 100 mL calibration standard/spiking solution is prepared as stated in the following table from vendor supplied primary standards or prepared in-house standards. The primary standards are stored in the standards refrigerator at 4° C. They are replaced annually. Upon receipt the bottles are logged into the standard logbook and are QC'd.

Analyte	Stock Conc. (mg/L)	Volume of Stock (µl)	Final Conc. (mg/L)
F	1000	500	4
Cl	1000	1000	10
NO_2 as N	1000	125	2
Br	1000	100	5
NO_3 as N	1000	100	4
PO_4 as P	1000	100	5
SO_4	1000	100	20

2.2.1.2 The above solution expires after three months. It is stored at room temperature. The routine calibration solution does not contain Br and PO_4. These analytes are added when required.

2.2.1.3 A 1000 mg/L bromide stock solution is prepared by diluting 0.149 g of desiccated NaBr to a final volume of 100 mL. This solution is prepared annually. Desiccated NaBr is prepared by first drying the salt in a 105°C ± 5°C drying oven for four hours. The salt is then cooled to room temperature in a desiccator.

2.2.1.4 A 1000 mg/L phosphate stock solution is prepared by diluting 0.439 g of desiccated KH_2PO_4 to a final volume of 100 mL. This solution is prepared annually. Desiccated KH_2PO_4 is prepared by first drying the salt in a 105°C ± 5°C drying oven for four hours. The salt is then cooled to room temperature in a desiccator.

2.2.1.5 Spiking - Samples are spiked by adding 100µl of solution prepared in 2.2.1.1 to an aliquot of sample and diluting to 500 µl. The dilution of sample and spike are recorded in the dilution column of the batch schedule. The reference number of the spike is recorded in the sample name field of the batch schedule.

2.2.2 ICV Solution

2.2.2.1 An ICV mix for fluoride, chloride, nitrite, bromide, nitrate, phosphate and sulfate is vendor purchased.

2.2.3 Eluant Solution

2.2.3.1 The eluant is 1.7 mM sodium carbonate and 1.8 mM sodium bicarbonate. The eluant is prepared by dissolving 0.170 g sodium bicarbonate and 0.740 g sodium carbonate in DIW and diluting to a final volume of 2 L.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.2 Waste Disposal

3.2.1 There is no significant waste generated from this procedure. Any remaining liquids are disposed of by flushing down the sink. Solid material may be disposed of in the garbage.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on the determination of method detection limits (LTL-1011). This SOP can be found in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration 1-5 times the anticipated method detection limit. A Student's t-test is then applied to these measured values to calculate the MDL.

4.1.2 MDL studies for the ion chromatograph must be determined at a minimum of every year months, with each new analyst, or whenever there is a significant change in system response.

4.1.3 Approximate MDLs

Analyte	Estimated MDL
Fluoride	0.02 mg/L
Chloride	0.04 mg/L
Nitrite	0.01 mg/L
Bromide	0.08 mg/L
Nitrate	0.01 mg/L
Phosphate	0.06 mg/L
Sulfate	0.08 mg/L

4.2 Determination of Retention Time Windows

4.2.1 Every six months, or any time there is a significant change in the system (such as analytical column replacement), the retention time windows need to be determined.

4.2.2 The RW is determined by taking the retention times of three CCVs (mid range standards) on any one day, and computing the standard deviation. The standard deviation multiplied by three is the retention time window. This procedure is similar to that described in the Laucks SOP on Determination of Retention Time Windows (LTL-8000) except one run is used rather than a 72-hour period. This value is then entered into the AI-450 method. The retention time windows are stored in the method.

4.2.3 This data is generated on an Excel spreadsheet and is archived on the laboratory data base.

4.3 Initial Multi-Point Calibration

4.3.1 A calibration curve consists of four points and a blank. All calibration standards are diluted in sample vials at the instrument just prior to analysis as stated in the following table:

Standard Number	Volume Standard	Volume DIW	Total Volume
1	25 µl	475 µl	500 µl
2	100 µl	400 µl	500 µl
3	250 µl	250 µl	500 µl
4	500 µl	0 µl	500 µl

Analyte	STD 1 (mg/L)	STD 2 (mg/L)	STD 3 (mg/L)	STD 4 (mg/L)
Fluoride	0.2	0.8	2	4
Chloride	0.5	2	5	10
Nitrite	0.1	0.4	1	2
Bromide	0.25	1	2.5	5
Nitrate	0.2	0.8	2	4
Phosphate	1	4	10	20
Sulfate	1	4	10	20

4.3.2 The elution order for this system is fluoride, chloride, nitrite, bromide, nitrate, phosphate, sulfate.

4.3.3 Samples whose responses exceed the high standard must be diluted.

4.3.4 A multi-point calibration of the system is not required with every analytical run.

4.3.4.1 If the previous calibration is verified by an ICV, the calibration is considered valid and the instrument does not require re-calibration.

4.3.4.2 A curve is not valid for a period of greater than six months.

4.3.5 Criteria

4.3.5.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The linear correlation coefficient (r) must be 0.995 or greater. All CCVs and sample concentrations must be computed using the regression equation.

4.3.6 Corrective action

4.3.6.1 If the criteria are not met the instrument must be recalibrated. No data may be reported for an analyte that does not have a valid calibration.

4.4 Initial Calibration Verification

4.4.1 At the start of each analytical run, analyze a standard from a source other than that from which the calibration material was obtained.

4.4.2 Criteria

4.4.2.1 The calculated concentration of the must be within the limits supplied by the manufacturer or not to exceed ±10% of the true value.

4.4.3 Corrective action

4.4.3.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated. No data can be reported for an analyte that does not have a valid ICV.

4.5 Initial Calibration Blank

4.5.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed the reporting limit.

4.5.2 Corrective action

4.5.2.1 If the ICB contains target analyte levels above ½ of the reporting the system is considered to be out of control. The source of contamination must be identified and corrected before proceeding with the analysis. No data can be reported for an analyte that does not have a valid ICB.

4.6 Continuing Calibration Verification (CCV) and Blank (CCB)

4.6.1 A mid-range calibration standard is analyzed at a minimum of once every 10 sample injections. Immediately following the CCV, a blank solution is analyzed. In addition, at the end of the run, this standard should be run at a different level than preceding CCVs to verify that the curve has remained linear throughout the analysis. A CCB should follow this final CCV.

4.6.2 Criteria

4.6.2.1 The CCV must fall within ± 10% of the true value for Method 300.0, and within ± 5% for Method 9056.

4.6.2.2 The levels of target analytes in the CCB should not exceed ½ of the reporting limit.

4.6.3 Corrective action

4.6.3.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful ICV and a CCV which is in control or by preceding and following CCVs which are within limits.

4.6.3.2 If the CCB contains target analyte levels above ½ of the reporting limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank that are in control or by preceding and following CCBs that are within limits.

4.7 Method Blanks

4.7.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time, or at least one blank every 20 samples, whichever is more frequent. Any analyte response above the reporting limit is reported. For the purpose of water analyses, the ICB, CCBs, and the method blank are the same. Method blank control limits are that contamination should not exceed ½ of the reporting limit for waters.

4.7.2 Corrective action

4.7.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.8 Matrix Spike/Matrix Spike Duplicate

4.8.1 Project-specific criteria will determine whether MS/MSD or MS/Duplicate analysis is to be performed. MS/MSD Duplicate precision is calculated as in Section 4.9 – precision usually is project-specific.

4.8.2 Samples are spiked by adding 100µl of calibration/spiking solution to an aliquot of sample and diluting to 500 µl. The dilution factor of the sample and the spike are

recorded in the dilution field of the batch schedule. The reference number for the spike is recorded on the bench sheet.

4.8.3 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS - S) * 100}{SA}$$

where: SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.8.4 The recovery criteria are detailed in the Quality Control Database and will change from time to time. The current control limits are:

Analyte	Lower Control Limit (%)	Upper Control Limit (%)	RPD Limit (%)
Fluoride	66	121	11
Chloride	73	121	11
Nitrite	50	150	30
Bromide	50	150	30
Nitrate	79	117	10
Phosphate	80	110	10
Sulfate	81	115	10

4.8.5 Corrective action

4.8.5.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also

look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.9 Sample Duplicate

4.9.1 Criteria

4.9.1.1 Sample duplicates are required when running Method 300.0 or 9056. At least one duplicate sample per 10 samples per matrix is required when method 9056 is being performed. At least one duplicate sample per 20 is required for Method 300.0. CLP protocols do not specify duplicate frequency and so the less-stringent criterion is used. RPD values are calculated in the following manner:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where: S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.9.1.2 The RPD control limits are detailed in the Quality Control Database and will change from time to time. The current control limits are detailed in 4.8.4.

4.9.2 Corrective action

4.9.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation Procedures

5.1 Initial Multi-Point Calibration

5.1.1 Creating a method

5.1.1.1 To start the Method Editor program double-click on the Method icon in the AI-450 MainMenu.

5.1.1.2 Method Settings:

System: Dionex DX-100
Number of Detectors: 1
Detector 1: Other-COND conductivity detector
1000mV=30.000uS
Run Time: 6-13 min. depending on retention time of sulfate peak
Plot Min.: -1
Plot Max: 30
Sampling Rate: 2 Hz
Timed Events: Init-Pump on
0.1:Offset on, Sampler on, Pump on, Begin Sampling on
Detector 1: Integration- Threshold-10.0, Width-10.0 sec, Reject-1000.0, Reference Peak area reject-2000.0
Data Events- void volume treatment for this peak at Fluoride retention time
Calibration- 4 levels, linear, calibration update- replace, standardization- external, Resp. for unknowns-area. The rest is set with the default settings.
Components- Enter each component that will be analyzed and their respective retention times, retention time windows. Calibrate by area. Reference component #1.
Enter amount of analyte at each calibration level.
Save new method as icMMDDYY.met where M=month, D=day, Y=year.

5.1.2 Creating a Calibration Curve Schedule

5.1.2.1 To start the Schedule Editor Program, double-click on the [Schedule] icon in the AI-450 MainMenu.

5.1.2.2 Enter sample name. For calibration purposes *autocal1r* is the name for the level 1 standard, *autocal2r* is the name for the level 2 standard, etc.

5.1.2.3 Enter the name of the method saved in 5.1.1.2

5.1.2.4 Enter a file name cMMDDYY, where M=month, D=day, Y=year. The program will number the filenames during analysis.

5.1.2.5 Save Schedule as cMMDDYY.sch., where M=month, D=day, Y=year.

5.1.3 Starting a run

5.1.3.1 Double-click on the [run] icon in the AI-450 MainMenu.

5.1.3.2 Load the schedule into the run program.

5.1.3.3 Load autosampler with cartridge containing the 4 levels of standards. Set autosampler to Run.

5.1.3.4 Set DX-100 to relay control.

5.1.3.5 Choose the run menu and start in the Run Program of AI-450.

5.2 Sample Analysis

5.2.1 Analysis tools and observations

5.2.1.1 If a discrete bottle number is referenced on the sample container, this information must be recorded in the sample name field of the batch schedule.

5.2.1.2 Samples that appear overly turbid or that have suspended or floating particles must be filtered through a 0.45 µm syringe filter prior to analysis. If this procedure is followed it should be documented.

5.2.1.3 If available, conductivity or TDS data can be used as a tool in determining appropriate dilutions.

5.2.1.4 Poor resolution between chloride and nitrite, phosphate and sulfate, and nitrate and bromide in standards or CCVs is an indication that column should be cleaned. If after cleaning the CCV does not resolve back to baseline between the two peaks of interest, the column has lost enough theoretical plates to render it unsuitable for analysis of those ions.

5.2.1.5 An abnormally shaped sulfate peak may indicate a sample with a high phosphate concentration. The chromatogram should be compared to one run at a greater dilution to see if acceptable peak resolution can be obtained.

5.2.1.6 The expected baseline conductivity for the system is 13.0 to 15.0 µS.

5.2.1.7 The expected back pressure of the system is 750 to 1600 psi. When the upper limit is approached the analyst should replace the eluant filter, replace the bed supports, clean the column, or reduce the flow rate. It is acceptable to reduce the flow down to 1.2 mL/min.

5.2.1.8 Other anomalies should be discussed with the departmental supervisor in order to determine appropriate corrective action.

5.3 Analysis sequence

Calibration standards (from low to high concentration)
ICV Solutions necessary for that analytical run
ICB
10 or fewer injections
midrange standard (CCV)
DIW (CCB)
additional samples (not more than 10 injections)
additional CCVs and CCBs
...
...
must close with CCV and CCB

5.4 Instrumental Conditions

5.4.1 System Conditions

- Eluant flow is between 0.5 and 2.5 mL/min.
- Injection volume is 500 µL.
- Temperature correction factor is 1.7.
- Detector range is 30 µS.
- Analysis time is between 10 and 20 minutes.
- Background conductivity is between 13-18 µS.

5.5 Analytical Operation

5.5.1 Analytical procedure for liquid matrixes

5.5.1.1 A set of samples is chosen to be analyzed. Consideration should be given to holding times, due dates, and analytes of interest when choosing the sample set for a run.

5.5.1.2 A schedule is constructed for the sample set. This includes the appropriate dilutions for the given samples, the method name that corresponds to the curve that is in effect, and the filename where data for the sample will be saved. The file name extension is updated during analysis, therefore the analyst will not need to type in an extension.

5.5.1.3 The schedule is loaded into the run program of AI-450 Chromatography Software.

5.5.1.4 An appropriate number of 500 µL sample vials are placed into the auto sampler trays.

5.5.1.5 500 µL of standards, samples and QC events are placed into the sample vials.

5.5.1.6 Dilutions up to 50x can be done directly in the sample vials, greater dilutions will require an external dilution in a Solo cup or a combination of external and sample cup dilutions.

5.5.1.7 Samples that appear turbid or that have suspended particles should be filtered through a 0.45 µm syringe filter prior placing in sample vials and/or dilution. If a sample is pre-filtered, it must be documented.

5.5.1.8 Sample vials are capped with filter caps and placed in the auto sampler.

5.5.1.9 The "Control" button on the DX-100 is set on relay and the autosampler is set on local control and run.

5.5.1.10 Choose the run menu and start in the Run Program of AI-450.

5.5.2 Analytical procedure for soil or sludge matrixes.

5.5.2.1 The soil/sludge is leached with DIW by transferring 10 g to a tared, 250 mL digest bottle, adding 100 mL DIW and placing on the shaker for 15 minutes or more at a shaking rate sufficient to suspend the majority of the solids. The solids are then allowed to settle and an aliquot of the supernatant is filtered through a 0.45 µm syringe filter prior to use. The leaching and filtration of the sample must be recorded in the pretreatment field of the batch summary and a prep sheet completed.

5.5.2.2 A 100 mL aliquot of DIW is shaken in a 250 mL digest bottle and used as a method blank.

5.5.2.3 The resulting supernatant is treated as a water sample.

5.5.2.4 A total solids determination is made on all soil/sludge samples and the results are reported on a dry weight basis.

5.6 Compound Quantification

5.6.1 The data acquisition system (AI-450 Chromatography Software, or equivalent) generates a report with the chromatogram of the sample, the date analyzed, the sample identification, the method used to quantify the sample, the sample dilution, and the calculated concentration of the sample corrected for dilution.

5.6.2 For soil/sludge samples, the mg/L integrator value is converted to mg/kg DB by the following equation

Soil:

$$Cs = \frac{Ci * Vs * 100}{Ws\ Ts}$$

Cs = Sample concentration (mg/kg DB)
Ci = Concentration in sample injected (mg/L)
Vs = Volume of extract (mL)
Ws = Weight of sample extracted (gm)
TS = Total solids (%)

5.6.3 The data is exported into a spreadsheet program (Excel or equivalent) to produce a sample results summary.

6. Reports

6.1 Sample Results and Quality Control Reports

6.1.1 Data packages shall consist of the sample results summary, the chromatograms for samples in the workorder, a copy of the calibration curve and standard log book, and a copy of the QC summary report form. These include the method blank analysis, matrix spike results, duplicate results, and the Standard Reference Material (SRM) reports. A copy of the QC summary report form must be included in order to indicate that the QC was evaluated for analytical consistency.

6.2 Data Qualifying Flags

6.2.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

Appendix I

Method 300.0A, 9056 QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	A minimum three point curve that brackets the sample concentrations and a blank.	A four point curve that brackets the sample concentrations Corr coef r≥ 0.995	As instrument performance dictates, or when any change is made in the analytical system.	Fix the problem and recalibrate.	In the raw data package for all validatable packages and referenced in all other data.
Initial Calibration Verification / SRM	Not to exceed ±10% of the true value.	Limits are vendor supplied but must not exceed ±10% of the true value.	Once per analytical run, or 1/20.	Reanalyze, if reanalysis fails recalibrate and or perform instrument maintenance. No data can be reported if ICV fails.	In the raw data package, and in the quality control data base.
Initial Calibration Blank	Value not to exceed the MDL.	Value not to exceed ½ of the Reporting Limit.	Immediately following the ICV.	Reanalyze. No data can be reported if ICB fails.	In the raw data package, and in the quality control data base.
Continuing Calibration Verification	Not to exceed ±10% of the true value.	Method 300.0: Not to exceed ±10% of the true value. ---------- - Method 9056 not to exceed ± 5% of the true value.	At a minimum, after every 10 samples and as the final injection.	Samples which are not bracketed by in control CCVs must be reanalyzed.	In the raw data package.
Continuing Calibration Blank	Value not to exceed the MDL.	Value not to exceed ½ of the Reporting Limit.	Immediately following a CCVs.	Samples which are not bracketed by in control CCBs must be reanalyzed.	In the raw data package.
Method Blank	Value not to exceed the MDL.	ICB and method blank are the same QA event for waters and must not exceed ½ of the reporting	Run at a minimum of 1 per 20 samples and as dictated	If sample concentration exceed 10x the contamination level, no action required.	In the raw data package, and in the quality control data base.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
		limit. Soil method blanks must not exceed ½ of the reporting limit.	by non-water matrixes.	Samples with contaminated Method Blanks must be re-prepared and reanalyzed.	
Matrix Spike /Matrix Spike Duplicate Recovery	Not to exceed ±20% of the true value or x ± 3S. Once every 10 samples.	To meet or exceed criteria in the quality control data base. Current limits are detailed in section 4.8.4.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.
Duplicate/ Relative % Difference (RPD)	NA	To meet or exceed criteria in the quality control data base. Current limits are detailed in section 4.8.4.	Method 300.0: Once every 20 samples, or per client request, whichever is more frequent. ---------- Method 9056: Once every 10 samples.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-9113

Title: **pH in Soil and Waste Samples Using SW846 Method 9045C**

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1.0	07/13/94
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UNCONTROLLED

Written by: Bill Lundberg Date: 1/16/97
Bill Lundberg, Inorganic Supervisor

Reviewed by: Harry Romberg Date: 1-16-97
Harry Romberg, QA Officer

Approved by: Karen J Kotz Date: 1/16/97
Karen Kotz, Lab Director

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is an electrometric procedure for the determination of pH in soils and waste samples.

1.1.2 The sample is mixed with deionized water (DIW). The pH of the solution is then measured with a pH meter.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Interferences:

1.2.1 Samples with very low or very high pH may give incorrect readings on the meter. For samples with a true pH of >10, which is not uncommon for some ash and other waste materials, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode. Strong acid solutions, with a true value of <1, an uncommon condition, may give incorrectly high measurements.

1.2.2 Temperature fluctuations will cause measurement errors. Most pH electrodes are calibrated to 25°C. Whereas Laucks does use a temperature correction probe with the pH meter, significant differences in pH can affect the solubility and apparent pH characteristics of the solution itself. Therefore, measurements should be made near the appropriate temperature by allowing the sample and diluent (DIW) to warm to room temperature prior to analysis.

1.2.3 Errors can occur when the pH electrode becomes coated. If this occurs, the electrode must be thoroughly clean and re-buffered prior to measuring the pH of subsequent samples.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 There is no official holding time for the analysis of soil pH. The holding time for aqueous samples is to analyze immediately. Soil should not demonstrate significant pH fluctuations if refrigerated and analyzed within a reasonable time but Laucks has chosen to analyze soils samples as soon as is reasonable after collection, not to exceed 7 days from collection.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.4.1.1 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.4.1.2 **Sequence** - A set of sample extracts and standard solutions presented to an instrument in a chronologically continuous group.

2. Equipment List, Reagents and Standards

2.1 Apparatus

pH meter with means for temperature correction (temperature correction probe)
Glass electrode
50 ml. beaker
Analytical balance capable of weighing to 0.1 g.

2.2 Standards

pH 4 buffer
pH 7 buffer
pH 10 buffer

2.3 Reagents

Deionized water (DIW)

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.2 Waste Disposal

3.2.1 There should be no significant waste associated with this procedure which cannot be disposed of in the laboratory sink and/or garbage.

4. Calibration and Quality Control

4.1 Calibration

4.1.1 The pH meter must be calibrated with at least two pH buffers. One is the pH 7 buffer and the other depends upon the samples. If high pH is expected, the second buffer should be at pH 10 or higher. If low pH is expected, the second buffer should be the pH 4 buffer. High pH samples should not be analyzed from a low pH calibration and visa versa.

4.2 Sample Duplicate

4.2.1 Criteria

4.2.1.1 At least one duplicate sample per 10 samples per matrix is required. RPD values are not currently calculated for this analysis. The control limit is considered to be 0.5 pH units.

4.2.2 Corrective action

4.2.2.1 If an out-of-control duplicate pair is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample preparation and pH measurement of soils

5.1.1 To 20 grams of soil in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.1.2 Allow the suspension to stand for about 1 additional hour with no agitation in order to allow most of the suspended particulates to settle out.

5.1.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the soil itself.

5.1.4 The electrodes should be thoroughly rinsed between samples.

5.1.5 Report the results as "soil pH measured in water @ ____ °C."

5.2 Sample preparation and pH measurement of waste material:

5.2.1 To 20 grams of waste material in a 50 ml beaker, add 20 mls of DIW and stir the suspension for 5 minutes. This may also be done with a magnetic stirring bar.

5.2.2 Allow the suspension to stand for about 15 additional minutes with no agitation in order to allow most of the suspended particulates to settle out.

> Note: If the waste is hygroscopic and absorbs all the DIW, begin the test again using 20 g of waste and 40 ml of DIW.
>
> Note: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous phase.

5.2.3 Immerse the electrode (including the temperature correction probe) just far enough into the solution to establish good electrical contact. It should not come into contact with the waste material itself.

5.2.4 The electrodes should be thoroughly rinsed between samples.

5.2.5 Report the results as "waste pH measured in water @ _____ °C.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. The report form must be filled out in order to indicate that the duplicate QC had been performed and met the criteria specified in this document.

Appendix I

QC Summary Table

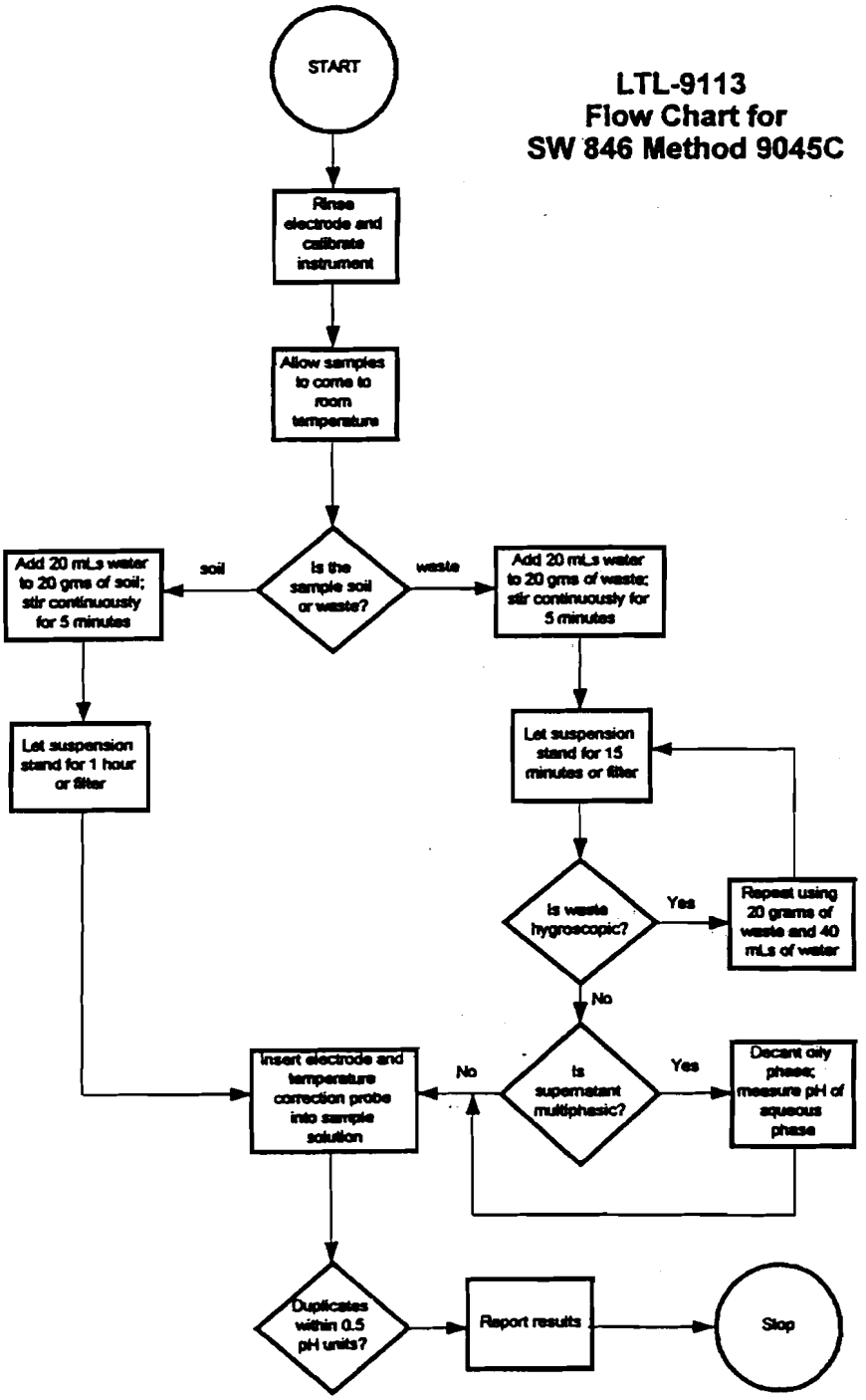
Laucks Testing Laboratories

Method SW 9045C QA Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	Buffer pH meter to bracket sample values	Buffer pH meter between pH 7 & 4 or 10 before analyses (to bracket sample values)	Once before analyzing any samples or QC	None	None
Duplicate % Difference	N/A	within 0.5 pH units of one another	10% or once every 10 samples.	Correct problem Consult QC Officer	QC_DB report form with appropriate commentary

Appendix II

Flow Chart


LTL-9113
Flow Chart for
SW 846 Method 9045C
START
Rinse electrode and calibrate instrument
Allow samples to come to room temperature
Is the sample soil or waste?
soil
waste
Add 20 mLs water to 20 gms of soil; stir continuously for 5 minutes
Add 20 mLs water to 20 gms of waste; stir continuously for 5 minutes
Let suspension stand for 1 hour or filter
Let suspension stand for 15 minutes or filter
Is waste hygroscopic?
Yes
Repeat using 20 grams of waste and 40 mLs of water
No
Is supernatant multiphasic?
No
Yes
Decant oily phase; measure pH of aqueous phase
Insert electrode and temperature correction probe into sample solution
Duplicates within 0.5 pH units?
Report results
Stop

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9115

Title: **The Determination of Total Organic Carbon in Water Samples by EPA 415.1 and SW846 Method 9060**

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Kathy Kreps, Laboratory Director

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1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used to determine the concentration of total organic carbon (TOC) in a water sample. The acidified sample is purged of inorganic carbon and then sent to the UV reactor where organic carbon is converted to CO_2 by UV-catalyzed persulfate oxidation. Released CO_2 is measured using NDIR detection.

1.1.2 Method SW846 9060 states that all TOC analyses should be performed in quadruplicate. Laucks performs all TOCs singleton, unless previous arrangements have been made with the client.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 It is strongly recommended that samples be collected in glass containers (40mL vials are provided by Laucks Testing Labs), preserved with 0.2% phosphoric acid. Samples collected in plastic run the potential risk of: a) an increased TOC value do to organic carbon leaching from the plastic into the sample and/or b) a decreased TOC value due to the solvents in the sample being incorporated into the plastic container.

1.2.2 In accordance with SW846, plastic containers are permissible. Laucks will, however, make every effort to provide glass containers.

1.2.3 Upon receipt, samples should be stored at 4 ± 2°C.

1.2.4 The maximum recommended holding time for waters is 28 days from the date of sample collection. If the holding time is exceeded, the analyst should notify the project manager so they may notify the client.

1.3 Interferences

1.3.1 This procedure is applicable only to homogenous samples which can be injected reproducibly into the apparatus. The opening of the syringe limits the maximum particle size which may be included in the sample.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as ms/msd or method blank, are not defined here. Since it is assumed that the user of this SOP already understands their more general meaning.

1.4.2 **CCB** - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline or is exhibiting carry-over from the CCV.

1.4.3 **CCV** - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.4.4 **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.4.5 **Corr Coef, CC, r** - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.4.6 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.4.7 **ICB** - Initial calibration blank - This term is borrowed from CLP. An instrument blank is prepared in the same way as calibration standards, without target analytes.

1.4.8 **ICV** - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate.

1.4.9 **MDL** - Method detection limit - The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are determined empirically by Laucks. See section 4.1 for a more detailed discussion of MDL determination.

1.4.10 **SRM or LCS** - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control.

1.4.11 **QC period** - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.

1.4.12 **RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.4.13 **Sequence**- A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Equpiment List and Standards

2.1 Equpiment List

2.1.1 Instrument

2.1.1.1 TOC analyzer, Tekmar-Dohrmann Phoenix 8000, or equivalent.

2.1.1.2 Autosampler

2.1.2 Analytical Balance: 0.1 mg sensitivity.

2.2 Reagents

2.2.1 21 % Phosphoric Acid Reagent: 188 mL DIW into rinsed bottle. Add 37 mL concentrated phosphoric acid (H_3PO_4). This reagent should be prepared monthly.

2.2.2 10% persulfate and 5% phosphoric acid: 213 mL DIW into rinsed bottle. Add 9 mL concentrated H_3PO_4 and add 25 g of high purity sodium persulfate ($Na_2S_2O_8$). This reagent should be prepared weekly.

2.2.3 Purified air, hydrocarbon free, 35 psi.

2.3 Standards

2.3.1 All standards should be preserved with 0.2% phosphoric acid.

2.3.2 **50 ppm dextrose ICV/SRM stock solution**: Dissolve 0.125 g of AR grade dextrose in DIW. Add a small amount phosphoric acid (H_3PO_4) for preservation and dilute to a final volume of one liter.

2.3.3 **10 ppm dextrose ICV/SRM working standard**: Weigh 8.0 g of the 50 ppm dextrose ICV/SRM stock solution into 40 mL vial and bring the total weight up to 40.0 g with DIW.

2.3.4 **2000 ppm potassium biphthalate (KHP) stock solution**: vendor supplied solution.

2.3.5 **40 ppm potassium biphthalate (KHP) stock calibration standard**: Dilute 20.0 mL of the 2000 ppm. KHP stock solution to 1 liter with DIW.

2.3.6 **20 ppm potassium biphthalate (KHP) calibration standard**: Dilute 20.0 g of the 40 ppm potassium biphthalate (KHP) stock calibration standard to 40.0 g with DIW.

2.3.7 **10 ppm potassium biphthalate (KHP) calibration standard**: Dilute 10.0 g of the 40 ppm potassium biphthalate (KHP) stock calibration standard to 40.0 g with DIW.

2.3.8 **5 ppm potassium biphthalate (KHP) calibration standard**: Dilute 5.0 g of the 40 ppm potassium biphthalate (KHP) stock calibration standard to 40.0 g with DIW.

2.3.9 **2 ppm potassium biphthalate (KHP) calibration standard**: Dilute 2.0 g of the 40 ppm potassium biphthalate (KHP) stock calibration standard to 40.0 g with DIW.

2.3.10 **1 ppm potassium biphthalate (KHP) calibration standard**: Dilute 1.0 g of the 40 ppm potassium biphthalate (KHP) stock calibration standard to 40.0 g with DIW.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully

grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.2 Waste Disposal

3.2.1 Waste products from this process are not considered harmful and are therefore collected at the instrument and disposed of down the sink.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP on MDL studies. Briefly, it involves the analysis of seven replicate samples spiked at a concentration near the anticipated method detection limit. A Student's t-test is then applied to these measured values to calculate the MDL. The LTL estimated MDL is 0.5 mg/L.

4.2 Initial Multi-Point Calibration

4.2.1 Analyze standard solutions using at least 5 different concentration levels.

4.2.2 Calibration Curve:

Concentration	Preparation
0 ppm standard	0.2% phosphoric acid
1 ppm standard	Described in 2.3.10
2 ppm standard	Described in 2.3.9
5 ppm standard	Described in 2.3.8
10 ppm standard	Described in 2.3.7
20 ppm standard	Described in 2.3.6

4.2.3 Analyze the standards from lowest concentration to highest concentration.

4.2.4 A multi-point calibration of the system is not required with every analytical run.

4.2.4.1 If the previous calibration is verified by an ICV, the calibration is considered valid and the instrument does not require recalibration.

4.2.4.2 A curve is not valid for longer than six months. It is recommended that a curve is run when any significant change in instrumentation, reagents, or standards occurs.

4.2.5 Criteria

4.2.5.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater for a 5-point calibration. All CCVs and sample extract concentrations must be computed using the regression equation.

4.2.6 Corrective action

4.2.6.1 If the criteria are not met, the instrument must be recalibrated.

4.3 Initial Calibration Verification

4.3.1 Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained.

4.3.2 Criteria

4.3.2.1 The calculated concentration of the ICV should not exceed 90%-110% of the true value.

4.3.3 Corrective action

4.3.3.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

4.4 Initial Calibration Blank

4.4.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed the reporting limit *(one-half the reporting limit for USACOE work).*

4.4.2 Corrective action

4.4.2.1 If the ICB contains target analyte levels above the reporting limit *(one-half the reporting limit for USACOE work)*, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

4.5.1 A mid-range calibration standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.5.1.1 The CCV must fall within ± 10% of the true value.

4.5.2 The levels of target analytes in the CCB should not exceed the reporting limit *(one-half the reporting limit for USACOE work)*.

4.5.2.1 Corrective action

4.5.2.2 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

4.5.2.3 If the CCB contains target analyte levels above the reporting limit *(one-half the reporting limit for USACOE work)*, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.6 Method Blanks

4.6.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. Any analyte response above the reporting limit is reported. Method blank control limits specify that contamination should not exceed the reporting limit *(one-half the reporting limit for USACOE work)*. In this case the Method Blank and the ICB are the same.

4.6.2 Corrective action

4.6.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the

associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Assurance Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.7 Matrix Spike/Matrix Spike Duplicate

4.7.1 Project-specific requirements will dictate whether MS/MSD or MS/Duplicate analysis is to be performed. Unless specified otherwise by the client, a sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery} = \frac{(SS - S) * 100}{SA}$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.7.2 Matrix Spikes are prepared by spiking a 40 g aliquot of sample with 200 μL of a 2000 mg/L KHP vendor supplied stock solution (equal to a 10 mg/L spike).

4.7.3 The recovery criteria are detailed in the Quality Control Database and will change from time to time. The current QC limit for recovery for MS/MSD is 70%-119%.

4.7.4 Corrective action

4.7.4.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.8 Matrix Spike Duplicate

4.8.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.8.2 RPD control limits are detailed in the Quality Control Database and will change from time to time. The current qc limit for RPD is 11%.

4.9 Sample Duplicate

4.9.1 Criteria

4.9.1.1 Sample duplicates are required only when the client requests them. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. When duplicate analysis is all that is being performed or when requested the frequency is 10%. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where:

S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.9.1.2 The RPD control limits are detailed in the Quality Control Database and will change from time to time. The current control limit for RPD is 11%.

4.9.2 Corrective action

4.9.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Ensure that there are ample amounts of reagents to complete an entire analysis.

5.1.2 It is recommended that the priming method then the cleaning method be run prior to calibration or sample analysis.

5.1.3 Analysis sequence

ICV/SRM
ICB/MB
Sample 1
Sample 1ms (or dup depending on client specifications)
Sample 1msd (or ms depending on client specifications)
Sample 2-8
CCV1
CCB1
Continue with samples with CCVs and CCBs every 10 injections.
Close with a CCV and a CCB.

Note: Samples are run in Singleton, unless clients specify otherwise.

5.2 Instrumental Conditions

5.2.1 The instrument should be in the ready mode for a minimum of two hours to allow for NDIR stabilization.

5.2.2 Gas flow rate should be around 200 cc/min.

5.3 Analytical Operation

5.3.1 Calibration Setup

5.3.1.1 Select Calibration Standards from the Setup Menu.

5.3.1.2 Save the calibration standards as MMDDYY where M=month, D=day, Y=year.

5.3.1.3 Select Calibration Set Active from the Setup Menu.

5.3.1.4 Select the calibration standards named as active in 5.3.1.2.

5.3.1.5 Select [Sample Setup] in the run screen.

5.3.1.6 Setup the Calibration Sequence according to 4.2.2.

5.3.1.6.1 To enter the appropriate standard Select TOC standard in the Sample Type column.

5.3.1.6.1.1 A window will open which allows selection of the appropriate standard.

5.3.1.6.1.2 This process is repeated for all standards.

5.3.1.7 The selected method should be TOC Range 0.1-20ppm C.

5.3.1.8 Make sure vial numbers correspond to the placement of the standards in the autosampler tray.

5.3.1.9 Set all standards to ready.

5.3.1.10 Select [Save/Use] to save the setup.

5.3.1.11 Select [Start] on the Run Screen.

5.3.1.12 After calibration is complete, a window will open. At least five standards should be selected for the calibration curve. Evaluate the curve based on the criteria outlined in 4.3.2.1.

5.3.2 Sample Setup

5.3.2.1 Place samples on the autosampler tray according to the sequence in 5.1.3.

5.3.2.2 Select [Sample Setup] in the run screen.

5.3.2.2.1 Entering QC samples

5.3.2.2.1.1 To enter QC samples (except for MS, MSD, and DUP) Select Calibration Verification in the Sample Type column.

5.3.2.2.1.2 A window will open which will allow for selecting the appropriate standard.

5.3.2.2.1.3 Repeat this process for all standards.

5.3.2.2.2 Entering Unknowns

5.3.2.2.2.1 Type the sample ID in the Sample ID column.

If a dilution is needed, type the dilution factor after the sample ID in the Sample ID column.

5.3.2.2.2.2 Select Sample in the Sample Type Column.

5.3.2.2.3 Set the method to TOC Range 0.1-20ppm C.

5.3.2.2.4 Ensure that the samples are set to ready and vial numbers are correct.

5.3.2.3 Select [Save/Use] to save the setup.

5.3.2.4 Select [Start] on the Run Screen to begin analysis.

5.4 Compound Quantification

5.4.1 Data export

5.4.1.1 Data can be exported into Excel.

5.4.1.1.1 Under the results menu, Select Multiple Analysis.

5.4.1.1.2 A window should open showing the results of the previous analysis.

5.4.1.1.3 Under the Edit Menu, Select Customize View in Excel.

5.4.1.1.4 Continue by following instructions that will appear.

6. Reports

6.1 Data Packet Organization

6.1.1 See Appendix I for a check list detailing data packet organization

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, and an MS/MSD or MS/duplicate report. Many analyses will also require an SRM, blank spike or other report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

7. Reference

USEPA Method for Chemical Analysis of Water and Wastes, Lastest Edition, Method 415.1

USEPA Test Methods for Evaluating Solid Waste, Latest Edition, Method 9060

Appendix I

Data Packet Organization

Excel Spreadsheet with Calculations
QC_DB form
Raw Data
Out of Control Form (if needed)

Appendix II

QC Summary Table

EPA Method 415.1 and Method SW846 9060 QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	per manufacturers instructions	Correlation Coefficient (r) of .995 or higher.	At least every six months or with any significant change in the system. Must be verified with an ICV.	Recalibration.	N/A
Initial Calibration Verification/ Standard Reference Material	verify with independent check standard	± 10% of True Value.	Once every 20 samples, or per client request, whichever is more frequent	Perform system maintenance and re-check the ICV/SRM. If the criteria still cannot be met, the system must be recalibrated.	N/A
Initial Calibration Blank/Method Blank	Not specified.	Not to exceed the reporting limit*(one-half the reporting limit for USACOE work)*.	Once every 20 samples, or per client request, whichever is more frequent	If sample concentration exceed 10x the contamination level, no action required. Samples with contaminated Method Blanks must be re-extracted.	Narrative comment in report in quality control data base, and in raw data package.
Continuing Calibration Verification	verify with an independently prepared check standard every 15 samples	± 10% of True Value.	After every 10 samples and at the end of analysis.	Check calculations or perform instrument maintenance. Recalibrate and reanalyze.	N/A
Continuing Calibration Blank	Not specified.	Not to exceed the reporting limit *(one-half the reporting limit for USACOE work)*.	After every 10 samples and at the end of analysis.	The source of contamination must be identified and corrected and the affected samples re-analyzed.	N/A
Matrix Spike Recovery	once every 10 samples; no criteria	To meet or exceed criteria in the quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.

QC Summary Table (cont.)

Method SW846 9060 QA Requirements and Corrective Actions

MS/MSD RPD	Not specified.	To meet or exceed criteria in the quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.
Duplicate % Difference	N/A	To meet or exceed criteria in quality control data base.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control data base, and in raw data package.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-9116

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FLOW CHART 22

1. Introduction and Scope

1.1 Method Description

1.1.1 This method is used to determine the concentration of total organic carbon in a soil or sediment sample. Inorganic carbon (carbonates) is removed by treatment of the homogenized sample with 1:1 nitric acid. The treated sample is then combusted in a furnace and the carbon dioxide evolved as a combustion product is measured using an infrared detector.

1.1.2 This method is based on SW846 method 9060, modified to accommodate the analysis of soils and sediments.

1.1.3 Assuming a 1:5 dilution factor, using 10 to 100 mg of diluted sample, the stated calibration curve allows for a concentration range of 0.1 to 2.0% carbon in the sample. This range can be extended in either direction by adjusting the sample dilution.

1.1.4 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples can be collected in glass or plastic containers. A minimum sample size of 50 grams is recommended.

1.2.2 Samples should be stored at 4°C ± 2°C. The holding time for soil samples is not established, but the holding time for water samples is 28 days from sample collection. For lack of other official guidance, 28 days from sample collection is used for the analytical holding time.

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.3.2 **CCV** - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.3.3 **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.3.4 **Corr Coef, CC, r** - Correlation coefficient - A measures of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.3.5 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.3.6 **ICB** - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

1.3.7 **ICV** - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard, which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate.

1.3.8 **IDL** - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal: noise ratio of least 3x. Used as a starting point for selecting MDL study spiking levels.

1.3.9 **MDL** - Method detection limit - The lowest concentration a sample that will yield a positive result that is greater zero at a known level of confidence. MDLs are determined empirically by Laucks.

1.3.10 **MDL standard** - Method detection limit standard - A standard prepared so that the concentrations of the target analytes are no greater than 4x the empirically determined MDLs. This standard is used to verify that the instrument or system is capable of detecting the target analytes on an ongoing basis.

1.3.11 **SRM or LCS** - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

1.3.12 **QC period** - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank

analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

1.3.13 **RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.3.14 **Sequence** - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

2. Interferences

2.1.1 The possibility exists for loss of organic compounds during the drying step. In addition, drying at a temperature lower than 105°C may not be adequate to remove all moisture (and entrained inorganic-carbon compounds) from the sample.

2.1.2 Due to the small sample aliquot size used for analysis it is important that the sample be homogenized. This step is particularly important for samples, which will be diluted using clean sand.

3. Equipment List and Standards

3.1 Equipment

3.1.1 TOC analyzer, Dohrmann Pheonix 8000 or equivalent

3.1.2 Dohrman 183 boat sampler or equivalent

3.1.3 Analytical Balance: 0.1 mg accuracy

3.1.4 Desiccator

3.1.5 Platinum combustion boats

3.1.6 Drying oven set at 70°C

3.1.7 Any laboratory PC equipped with Excel and the TOC template may be used for calculation or the calculations may be done by hand, requiring no computer except for entry into the LIMS database.

3.2 Reagents and Standards:

3.2.1 Nitric acid (HNO_3), AR-Grade - Prepare by diluting 50 mL concentrated HNO_3 to 100 mL with DIW.

3.2.2 ASTM Type II water

3.2.3 Potassium hydrogen phthalate (KHP)Stock Solution, 4000 mg/L carbon: Dissolve 1510 mg ±1 mg KHP (Primary Standard Grade, dried for two hours at 120°C and stored in a desiccator) in a 200 mL volumetric flask. Calibration solutions are prepared from the stock by dilution. They must be stored under refrigeration and preserved with 0.2% H_3PO_4 and expire after one year.

3.2.3.1 Prepare solutions at 500, 1000, 1500, and 2000 mg/L as follows:

Analyte	Stock Conc.	Vol./Wt. taken	Final Vol.	Final Conc.	Solvent
500 mg/l KHP	4000 mg/l	12.5 ml	100 ml	500 mg/l	0.2 % H_3PO_4
1000 mg/l KHP	4000 mg/l	25.0 ml	100 ml	1000 mg/l	0.2 % H_3PO_4
1500 mg/l KHP	4000 mg/l	37.5 ml	100 ml	1500 mg/l	0.2 % H_3PO_4
2000 mg/l KHP	4000 mg/l	50.0 ml	100 ml	2000 mg/l	0.2 % H_3PO_4

3.2.4 Sand (purified and ignited, carbon-free), which has been disk pulverized to pass at least 80 mesh.

3.2.5 Laboratory Control Sample (LCS) is prepared as a 1:20 dilution in clean sand using Reference material ERA Sludge (currently, TV = 2.61%, range = 2.09 - 3.03% as prepared, Lot 17095) or equivalent. The control limits for this and any replacement materials are located in the laboratory's quality control database.

3.2.6 Matrix spike samples are prepared using 40 µL of the 1000 mg/L KHP working standard. Recovery limits are generated periodically as described in Laucks' SOP LTL-1011 and are located in the laboratory's quality control database.

4. Safety precautions and Waste Disposal

4.1 Safety Precautions

4.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

4.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

4.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

4.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

4.1.5 The combustion furnace is heated to approximately 800°C. Care should be taken not to burn oneself when in the proximity of the furnace or objects, which have been recently removed from the furnace.

4.2 Waste Disposal

4.2.1 There is no significant waste generated from this procedure. Any remaining liquids are disposed of by flushing down the sink. Solid material (ash, soil, etc.) may be disposed of in the garbage.

5. Calibration and Quality Control

5.1 Batch Quality Control Limits

5.1.1 All Batch QC precision and recovery criteria are maintained and updated in the Laboratory's Quality Database, which is managed by the Quality Assurance Officer. These criteria should be consulted prior to initiating sample analysis. This includes detection and quantitation/reporting limits and all associated control charts (see LTL-1011 and section 7.2 for control chart discussion).

5.2 Method Detection and Reporting Limits

5.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP LTL-1011, which is in turn derived from 40CFR part 136, Appendix B. Briefly, it involves the analysis of 7 replicate samples

spiked at a concentration near the anticipated method detection limit. A Student's t-test is then applied to these measured values to calculate the MDL.

5.2.2 The MDL has historically been no higher than 0.02% based on the standard sample size, and is usually lower. The Army Corps Shell document allows reporting of non-detects (ND) at a level no lower than about twice the MDL and requires flagging of data between that and the quantitation limit (low standard). The laboratory has selected a standard reporting limit of 0.1% to allow for the fluctuation of MDLs and for the slight background TOC expected from the method and diluent sand, which can be controlled but never completely eliminated. A low standard at this level is analyzed with each run to verify its acceptability. The Shell allows for the reporting of flagged results between this low standard and twice the MDL but the laboratory rarely will report any values below the reporting limit represented by this low standard.

5.3 Initial Multi-Point Calibration

5.3.1 The initial, multi-point calibration should be done as needed. A multi-point calibration is not required with each analytical run. If the previous calibration is verified by an ICV, the calibration is considered valid and the instrument does not require recalibration.

5.3.1.1 A curve is not valid for longer than six months.

5.3.2 Calibration Curve

Concentration of KHP	Injection Volume
2000 mg/L	40 µl
1500 mg/L	40 µl
1000 mg/L	40 µl
500 mg/L	40 µl
0 mg/L DIW	40 µl

5.3.3 Calibration Setup

5.3.3.1 Select Calibration Standards from the Setup Menu.

5.3.3.2 Save the calibration standards as MMDDYY where M=month, D=day, Y=year.

5.3.3.3 Select Calibration Set Active from the Setup Menu.

5.3.3.4 Select the calibration standards that you named in 5.3.3.2 as active.

5.3.3.5 Select [Sample Setup] in the run screen.

5.3.3.6 To enter the appropriate standard Select TOC standard in the Sample Type column.

5.3.3.6.1 A window will open which will allow for choosing the appropriate standard.

5.3.3.7 The selected method should be TOC Range 0.1-20ppm C.

5.3.3.8 Select [Save/Use] to save the setup.

5.3.4 A small piece of quartz wool is placed into the boat. This should be replaced daily. A 40 µl aliquot of the 2000 mg/L standard is injected onto the quartz wool.

5.3.5 Press the "start" button, and slide the boat smoothly into the furnace at about two inches per second.

5.3.6 Repeat this procedure with each of the other calibration standards (1500, 1000, and 500 mg/L). In addition, analyze a calibration blank (DIW) just after the last calibration standard. Report this blank as a sample with no dilution.

5.3.7 Criteria

5.3.7.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater for a 4-point calibration. All CCVs and sample extract concentrations must be computed using the regression equation.

5.3.8 Corrective action

5.3.8.1 If the criteria are not met, the instrument must be recalibrated.

5.4 Initial Calibration Verification

5.4.1 At the start of each analytical run a mid-range standard (1000 ppm C) is analyzed to evaluate the validity of the calibration curve.

5.4.2 Criteria

5.4.2.1 If the ICV is in control, the previous calibration is still valid. A new calibration must be performed when the ICV is out of control. It should read ±10% of the "true" value (between 900 and 1100 mg/L).

5.4.3 Corrective Action

5.4.3.1 Analysis of actual samples should not be started unless calibration of the instrument has been verified. If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated.

5.4.3.2 **No sample values should be reported from any run where the calibration has not been adequately verified.**

5.5 Initial Calibration Blank

5.5.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed 1/2 of the reporting limit.

5.5.2 Corrective action

5.5.2.1 If the ICB contains target analyte levels above one half of the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

5.6 System Blank

5.6.1 Prior to the analysis of any samples, a system blank is analyzed to ensure that the sample boat is free from contamination

5.6.2 Criteria

5.6.2.1 The system blank must read ≤2 ppm at the instrument. If the system blank reads higher than 2 ppm, the source of contamination must be identified and corrected before proceeding with the analysis.

5.7 Method Blanks

5.7.1 Since there is not a "method blank" in this method the diluent blank will be used as a method blank to estimate the level of 'background' contamination. See the diluent blank discussion in section 5.9.

5.8 Field rinsates, field blanks

5.8.1 Criteria

5.8.1.1 Field rinsates and blanks are analyzed only upon client request and are not a routine part of the method. These aqueous samples are analyzed in a manner similar to calibration standards. That is, 40 µl of the aqueous sample is injected onto quartz wool in a combustion boat and ignited. The measured TOC value is computed assuming a 40 mg sample size with no dilution.

5.8.1.2 The TOC content cannot exceed the reporting limit.

5.8.2 Corrective action

5.8.2.1 Reanalyze the affected field QC sample. Notify the client project manager immediately since all samples from the set are potentially adversely affected.

5.9 Diluent Blank

5.9.1 Purified and ignited, carbon-free sand is to be used as the sample diluent. This sand must be confirmed to have carbon content less than the reporting limit for the analysis (0.1%) or produce a reading of less than 5% of the level in any sample for which results are reported. If a measurement of the diluent sand records detectable levels of organic carbon, a minimum of three replicates will be required to be analyzed. The resulting quantitation and reporting limits may be adjusted to reflect this mean detectable level, if necessary, but the routine reporting limit of 0.1% should be adequate to allow for low level contamination without reporting false positive sample results or results with a significant bias.

5.10 Method Detection Limit Standard

5.10.1 No method detection limit standard is utilized for evaluation in this analysis. However, the low standard is very near the reporting limit and must meet the calibration criteria discussed in the calibration section of this method.

5.11 Continuing Calibration Verification (CCV)

5.11.1 Criteria

5.11.1.1 A mid-range (1000 mg/L) calibration standard is analyzed after every 10 samples. In addition, this standard must be the last sample analyzed in the run.

5.11.1.2 The CCV must fall within ± 10% of the true value.

5.11.2 Corrective action

5.11.2.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV, which is in control or by preceding and following CCVs, which are within limits.

5.12 Continuing Calibration Blank (CCB)

5.12.1 Immediately following the analysis of the CCV standard, a CCB must be analyzed. The CCB is 40 µL of DIW injected directly into the sample boat. The levels of target analytes in the CCB should not exceed 1/2 of the reporting limit.

5.13 Matrix Spike

5.13.1 Criteria

5.13.1.1 A sample is chosen at random from the samples to be analyzed, unless a client has specifically requested that a certain sample be spiked. An appropriate aliquot (generally 40µL) of the 1000 mg/L calibration solution is added to this sample prior to the combustion step. This addition should be at least double the instrument response. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of TOC. The TOC recovery is calculated as follows:

$$\%recovery = \frac{(SS - S)}{S_{true}} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
S_{true} = actual ("true") concentration of spike added

5.13.1.2 The recovery control limits are defined in the QC database. The current QC limit for MS/MSD is 50%-150%.

5.13.2 Corrective action

5.13.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the SRM analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

5.14 Sample Duplicate

5.14.1 Criteria

5.14.1.1 At least one duplicate sample per 20 samples per matrix is required. The same sample selected as the matrix spike sample is also selected for the duplicate analysis unless the client has specifically requested that another sample be used.

5.14.1.2 RPD values are calculated as follows:

$$RPD = \frac{|S_1 - S_2|}{(S_1 + S_2)/2} * 100$$

where:
S_1 = measured concentration in the initial analysis
S_2 = measured concentration in the duplicate analysis

5.14.1.3 The RPD control limit is defined in the QC database. The current QC limit for RPD is 20%.

5.14.2 Corrective Action

5.14.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

5.15 Standard Reference Material

5.15.1 Criteria

5.15.1.1 The standard reference material (SRM) must be analyzed at a frequency of once per 20 samples or per analysis batch whichever is more frequent.

5.15.1.2 The measured TOC value must be within the limits established by the SRM vendor. Alternatively, these limits may be unachievably narrow and may be

defined internally by the QA Officer. Acceptance limits are currently, TV = 2.61%, range = 2.09 - 3.03% as prepared, for ERA Lot 17095. The control limits for this and any replacement materials are located in the laboratory's quality control database.

5.15.2 Corrective Action

5.15.2.1 Appropriate corrective actions may include any or all of the following: check calculations, perform instrument maintenance, recalibrate the instrument, and reanalyze all affected samples. Corrective actions must be accepted by the QA Officer before data may be reported.

5.15.2.2 In any case, the SRM results must be recorded in the QC database and any corrective actions noted.

6. Operation procedures

6.1 Instrumental Conditions

6.1.1 The instrument with boat inlet is assembled according to the manufacturer's instructions.

6.1.2 A gas flow of 200 mL/min O_2 is set for the furnace.

6.1.3 Analysis sequence

6.1.3.1 Calibration standards (including calibration blank)

Aqueous ICV
Aqueous ICB
System Blank
Sand blanks (at least 1 per batch in triplicate)
Reference Material
PBS
10 or fewer samples (may include QC)
midpoint calibration standard (CCV)
calibration blank (CCB)
additional samples (no more than 10)
additional CCVs and CCBs
...
must close with CCV and CCB

6.2 Sample Analysis

6.2.1 Sample preparation

6.2.1.1 It is critical that each sample be thoroughly homogenized before a subsample is taken for analysis. If the sample is already finely divided with no large pieces, a homogeneous sample of 5 to 10 grams is dried in a 70°C oven. If samples contain larger pieces or if they in any way may compromise the analyst's ability to obtain a representative sample, the analyst should utilize sample size of at least 20 grams.

6.2.1.2 If the sample is already finely divided as described above, the dried sample, if it has formed clumps in the drying process, may be ground with a mortar and pestle to pass an 80 mesh sieve.

6.2.1.3 If the sample is sandy, rocky, or as described above, required a larger sample size; it should be ground using the disk pulverizer.

6.2.1.4 Following the grinding step, mix the ground sample thoroughly and combine a weighed, representative portion with clean diluents sand in a ratio of 1 part by weight sample to 4 parts by weight of sand. Record the weights of both sample and sand used. The mixture is placed in a snap-cap vial and tumbled for 90 minutes in the TCLP extractor. Dried, pulverized samples should be held in a desiccator.

6.2.2 Instrument startup

6.2.2.1 The furnace is turned on and the temperature allowed stabilizing. This takes approximately 30 minutes.

6.2.2.2 The instrument is set to the Boat Sampler Mode.

6.2.2.3 Sample combustion

6.2.2.4 Just prior to the combustion analysis, a sample aliquot (20 - 100 mg) is weighed into a combustion boat and wetted with 1:1 HNO_3. The boat is then placed in the 70°C oven and excess HNO_3 driven off (not more than 15 minutes).

6.2.2.5 The sample ID is entered into the sample setup screen and saved.

6.2.2.6 Insert a combustion boat containing treated sample into the wire loop. Close the lid and allow the instrument baseline to stabilize. This normally takes about two minutes.

6.2.2.7 Press the "start" button, and slide the boat smoothly into the furnace at about two inches per second.

6.2.2.8 After the instrument indicates the analysis cycle is complete, remove the boat, and after stabilization of the system, analyze the next sample in the sequence.

6.2.2.9 The same procedure is followed for blanks, samples, SRMs, and matrix spike samples. The size of the platinum boat and the percent carbon in the samples are the limiting factors for optimal sample size. Ten to 100 milligrams of prepared samples are used for analysis, with 40 mg preferred.

6.2.2.10 Total analysis time is approximately 5-6 minutes.

6.2.2.11 In some cases it may be necessary to analyze samples in quadruplicate. Check the LIMS comments to determine whether this is necessary.

6.3 Quantification

6.3.1 Micrograms sample burned

$$\mu g \text{ of sample burned} = \frac{(Ws*100)*Wb*1000}{(Ws+Wd)}$$

where:

W_s = weight (gm) sample diluted with sand
W_d = weight (gm) diluent sand used in sample preparation
W_b = weight (mg) sample combusted

6.3.2 Milligrams sand burned

$$\text{mg sand burned} = \frac{W_d}{W_s + W_d} * W_b$$

6.3.3 "sand correction"

$$C_s = \text{mg sand burned } (W_b) * \text{Corr}$$

where:

C_s = sand correction (response units)
Corr =correction factor for sand which is calculated by taking the average of three consecutive sand blanks.

6.3.4 Net sample reading

$$R_S = R - C_S$$

where:

R_S = net sample reading (response units)
R = uncorrected sample reading (response units)

6.3.5 TOC result

$$\% \text{ TOC} = \frac{R_S * CF * 100}{\mu g \text{ sample burned}}$$

where:

CF = calibration factor. The calibration factor is 0.04 because the instrument adjusts the result for calibration.

7. Reports

7.1 Sample Results and Quality Control Reports

7.1.1 In addition to the sample results, all results for quality control tests are entered into the lab database using the QC Database program. These results include those for the method blank analyses, matrix spike results, duplicate results, and Standard Reference material (SRM) reports. It is not necessary to include with each job a copy of the information entered into the QC Database program. However, for analytical consistency, a copy of the QC Database Report form MUST be included in order to indicate that the QC was evaluated.

7.1.2 Optional reports, which may be provided on client request, include initial and continuing calibration information and raw data.

7.2 Control Chart(s)

7.2.1 The recovery values for the LCS/SRM are plotted on control charts, which also may be supplied to clients upon request. Control limits are computed as discussed in Laucks SOP LTL-1011, "Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies and Control Limits". Basically, warning limits are established as twice the standard deviation of the mean of at least 20 data points and control limits as 3 times the standard deviation. If it is a purchased laboratory control material for which the vendor has computed limits, those limits are used for control purposes. Control charts are developed and interpreted in accordance with section 7.6 of Laucks Quality Assurance Plan. Essentially, a method has exceeded

control when any point exceeds a control limit, when 3 consecutive points have exceeded a single warning limit, 8 or more points are on one side of the mean, or a pattern of increasing or decreasing points or cyclical pattern is readily observable.

7.3 Definitions:

CODE	Definition
U:	The analyte of interest was not detected, to the limit of detection indicated.

7.4 Data Review

7.4.1 Data validation is detailed in a separate SOP (see LTL-9001)

8. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, USEPA, 1986, Method 9060.

Appendix I

Laucks Testing Laboratories

Method SW 9060 (modified for soil/sediment) QA Requirements and Corrective Actions

QA Element	Method Criterion (for water)	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	per manufacturers instructions	4-point curve. 0.995 or higher correlation coefficient (r)	As needed. Expires after six months.	Fix problem and re-calibrate	in raw data package
Initial Calibration Verification	verify with check standard	Verify the calibration with 1000 mg/L KHP solution	At the beginning of each run	prepare new ICV and/or calibration solutions; service instrument; report no analyses from failed ICV	in raw data package
Initial Calibration Blank	Analyzed immediately after ICV	Must be less than ½ the reporting limit	Analyzed immediately after ICV	Source of contamination must be identified and corrected before proceeding with analysis	in raw data package
Continuing Calibration Verification	verify with an independently prepared check standard every 15 samples	verify with a mid-point standard; CCV must fall within 10% of the true value	after every 10 samples	Fix problem & recalibrate; re-analyze samples which are not bracketed by in-control CCVs	in raw data package

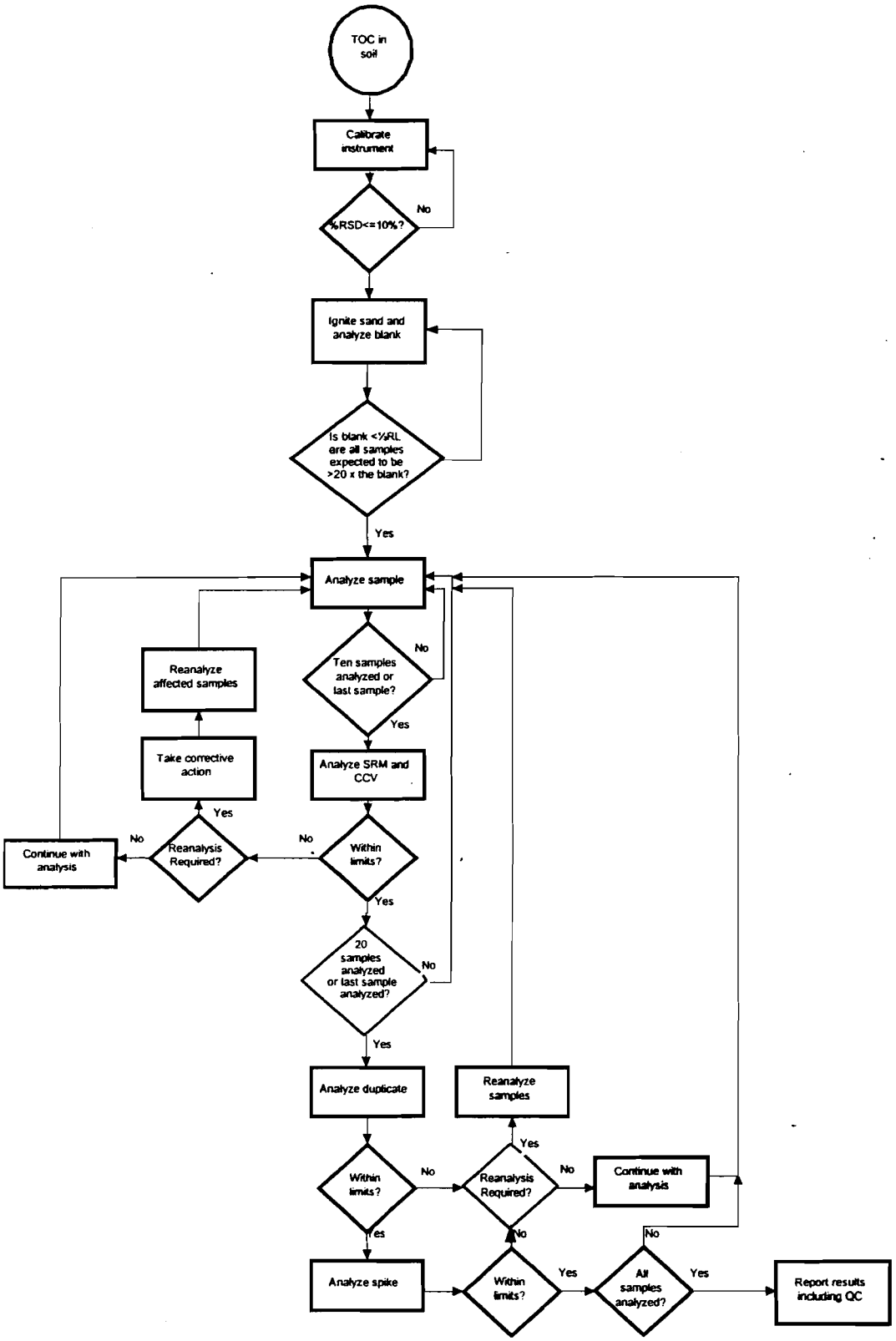
QA Element	Method Criterion (for water)	Laucks Criterion	Frequency	Corrective Action	Documentation
Continuing Calibration Blank	Analyzed immediately after CCV	Must be less than ½ the reporting limit	Analyzed immediately after CCV	Source of contamination must be identified and corrected. Affected samples must be reanalyzed.	in raw data package
Reporting Limit standard recovery	N/A	Must meet initial calibration criteria	Reporting limit standard is incorporated into curve	Recalibrate	in raw data package
System Blank	N/A	Must be ≤2 ppm instrument reading	Once after initial calibration	Check system for leaks; check sample introduction technique; do not analyze samples	in raw data package
Diluent Blank/Method Blank (sand blank)	Once per sample batch; no criteria	Diluent (sand) blank is determined and must be < ½ RL or 5% of any reported sample.	Once per batch or per twenty samples, in at least triplicate	If blank is very high, diluents sand should be re-ignited	in raw data package
Matrix Spike Recovery	once every 10 samples; no criteria	50% - 150% unless criteria are published in lab QC database	once every 20 samples or per batch, whichever is more frequent	Review data; Consult QC Officer; some conditions may require re-analysis	narrative comment in report and raw data package

QA Element	Method Criterion (for water)	Laucks Criterion	Frequency	Corrective Action	Documentation
Duplicate % Difference	N/A	20% unless criteria are published in lab control limits catalog or QC database	once every 20 samples or per batch, whichever is more frequent	Review data; Consult QC Officer; some conditions may require re-analysis	narrative comment in report and raw data package
Standard Reference Material (SRM) Recovery	N/A	ERA Sludge	once every 20 samples or per batch, whichever is more frequent	Review data; Consult QC Officer; some conditions may require re-analysis	narrative comment in report and raw data package

Appendix II

Flow Chart

TOC Analysis of Soils
by modified SW 846 Method 9060



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9136

Determination of Perchlorate by Ion Chromatography by EPA Method 314.0

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METHOD 314.0 QC REQUIREMENTS AND CORRECTIVE ACTIONS 22

1. Introduction and Scope

1.1 Method Description

1.1.1 This method describes the technique used to determine perchlorate in surface water, ground water and drinking water using Ion Chromatography. The method is capable of being extended to the analysis of perchlorate in soil matrices if a preliminary leaching step is performed to solubilize the perchlorate

1.1.2 The anions in an aqueous solution are introduced via an autosampler into a flowing eluant stream. Perchlorate is separated by an analytical column. The separation column effluent is then passed through an anion micromembrane suppresser where counter ions are exchanged for hydronium ions. This serves to suppress the conductance of the eluant and enhances the conductance of the perchlorate by forming its complementary acids. Following this exchange the stream is passed through a conductivity detector. The detector output is collected by a data acquisition system, which stores the chromatograms for later calculation and interpretation.

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1 Samples are collected in 500 or 1000 mL unpreserved plastic or glass bottles. Samples do not need to be shipped iced or stored cold in a refrigerator, but care should be taken not to expose samples to temperature extremes. No preservation is required.

1.2.2 Analysis holding time is 28 days.

1.3 Interferences

1.3.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

1.3.2 Pretreatment cartridges are effective in eliminating certain matrix interferences. Extreme caution should be exercised when using pretreatment cartridges. Artifacts can leach from some cartridges which can cause damage to the columns resulting in a loss of

column capacity. Cartridges should be washed with DIW according to manufacturer's instructions prior to use.

1.3.3 Sample matrices with high concentrations of anions such as chloride, sulfate, and carbonate can cause the baseline to destabilize at the retention time of perchlorate. Sample conductivity is monitored prior to analysis. When the Matrix Conductivity Threshold (MCT) is exceeded, the samples are pretreated to reduce the concentration of the common anions or diluted to reduce interference.

1.3.4 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in IC.

1.3.5 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

1.4 Definition of Terms

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline and to estimate carry-over from a mid-level standard (CCV).

CCV - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration. The method refers to the CCV as the Continuing Calibration Check Standard (CCCS)

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

Corr Coef, CC, r - Correlation coefficient (r)- A measure of the "goodness of fit" of a set of data to a regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is within control. The method refers to the ICV as the Initial Calibration Check Standard (ICCS)

MDL - Method detection limit - The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are determined empirically by Laucks. See section 4.2 for a more detailed discussion of MDL determination.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. May also be referred to as a blank spike. The method refers to the LCS as the Laboratory Fortified Blank (LFB).

QC period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

RT - Retention time - The time (in minutes) at which a target analyte elutes from the IC (or other) column.

RT window - Retention time window - The +/- value which is applied to the individual analyte peaks to establish the time range used to make tentative target analyte identifications.

Sequence - A set of sample extracts and standard solutions injected into an instrument in a chronologically continuous group. See also QC period.

MCT – Method Conductivity Threshold – The highest conductance of a sample, measured prior to analysis, which is used to determine whether a sample should be pretreated or diluted prior to analysis to reduce interferences from common anions.

IPC – Instrument Performance Check Solution – A solution containing a specific concentration of perchlorate and other common anions (chloride, sulfate, and carbonate) used to evaluate the performance of the instrument with respect to a defined set of criteria.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Ion chromatograph, Dionex model DX100 or equivalent consisting of a single piston pump, autosampler, either manual or automated injector, guard column, separator column, anion micromembrane suppresser, and a conductivity detector.

2.1.2 Guard column, Dionex AG16 or equivalent

2.1.3 Chromatographic column, Dionex AS16 or equivalent

2.1.4 Anion Micro Membrane Suppresser, Dionex ASRS-1 or equivalent. *Note: If pretreated sample are analyzed regularly, then cations from the cartridges may bind to the suppressor resulting in reduced peak response or asymmetrical perchlorate peaks. The suppressor should be cleaned regularly according to the manufacturer's instructions.*

2.1.5 Electronic data collection device, AI-450 Chromotography Software or equivalent, or other applicable data collection system.

2.1.6 Conductivity Meter

2.1.7 Analytical Balance

2.1.8 Plastic disposable syringes

2.1.9 Matrix pretreatment cartridges

2.1.9.1 Ba – reduce the matrix levels of sulfate

2.1.9.2 Ag – reduce the matrix levels of chloride

2.1.9.3 H - reduce Ag that may have leached from the Ag cartridge which may damage the suppressor. May indirectly reduce the levels of carbonate by removing the cationic counter ion.

2.2 Standards and Solutions

2.2.1 Perchlorate calibration primary stock solution (1000 mg/L): Dissolve 0.1231 g sodium perchlorate ($NaClO_4$) in DIW and dilute to 100 mL in a volumetric flask.

2.2.1.1 The above solution expires after one year. It may be stored at room temperature.

2.2.1.2 Spiking - Samples are spiked by adding 100µl of solution prepared in 2.2.1.1 to an aliquot of sample and diluting to 500 µl. The dilution of sample and spike are recorded in the dilution column of the batch schedule. The reference number of the spike is recorded in the sample name field of the batch schedule.

2.2.2 Perchlorate calibration secondary stock solution (1.0 mg/L): Dilute 0.5 mL of the perchlorate calibration primary stock solution to a final volume of 500 mL in a volumetric flask. This solution expires after one month. It may be stored at room temperature.

2.2.3 Perchlorate calibration solution #1 (400 µg/L): Dilute 80 mL of perchlorate calibration secondary stock solution to 200 mL in a volumetric flask. This solution expires after one month. It may be stored at room temperature.

2.2.4 Perchlorate calibration solution #2 (25 µg/L): Dilute 0.625 mL of the perchlorate calibration secondary stock solution to a final volume of 25 mL in a volumetric flask. This solution expires after one month. It may be stored at room temperature.

2.2.5 Mixed common anion stock solution: Dissolve the following salts in DIW to a final volume of 25 mL in a volumetric flask:

1.0 g sodium chloride (NaCl)
0.93 g sodium sulfate (Na_2SO_4)
1.1 g sodium carbonate (Na_2CO_3)

2.2.5.1 Each anion concentration in this solution is 25 mg/mL. It is used to prepare simulated common anion samples in the determination of the MCT.

2.2.5.2 The above solution expires after one year. It may be stored at room temperature.

2.2.6 MCT standards: Prepare the following seven standards in separate 25 mL volumetric flasks. Before bringing to volume, add 0.625 mL of the perchlorate calibration secondary stock solution.

Volume (mL) of mixed common anion stock solution	Measured conductivity range (µS/cm at 25°C)
0.20	1350-1500
0.30	1900-2100
0.40	2600-2800
0.50	3200-3700
0.60	3700-4000
0.80	4800-5200
1.00	5900-6400

2.2.7 Instrument performance check standard: a standard containing sulfate, chloride, carbonate and 25 µg/L of perchlorate at which the conductivity is +/-10% of the determined MCT. Confirm the conductance of this standard prior to analysis. If the conductivity has changed by more than 10%, a fresh standard must be prepared.

2.2.8 Perchlorate ICV stock solution (1000 mg/L): Dissolve 0.1393 g potassium perchlorate in DIW and dilute to 100 mL in a volumetric flask. This solution expires after one year. It may be stored at room temperature.

2.2.9 Perchlorate ICV standard (80 µg/L): Dilute 0.080 mL of Perchlorate ICV stock solution to 1 L in a volumetric flask. This solution expires after one year. It may be stored at room temperature.

2.2.10 Conductivity meter calibration solution: Dissolve 0.745 g potassium chloride (KCl) in DIW and dilute to a final volume of 1.0 L in a volumetric flask. The conductance of this solution is 1410 µS/cm at 25°C.

2.2.11 Eluant Solution

2.2.11.1 The eluant is 50 mM sodium hydroxide solution. The eluant is prepared by dissolving 8.0 g of 50% sodium hydroxide in DIW and diluting to a final volume of 2 L. Exposure to the atmosphere should be minimized to reduce possible carbonate contamination. Should be prepared fresh weekly and purged for 10 minutes with helium prior to use. The suppressed conductivity background of this solution should be between 2–5 µS.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.2 Waste Disposal

3.2.1 There is no significant waste generated from this procedure. Any remaining liquids are disposed of by flushing down the sink. Solid material may be disposed of in the garbage.

4. Calibration and Quality Control

4.1 Minimum Conductivity Threshold Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish the minimum conductivity threshold. It involves preparing a series of standards with increasing amounts of common anions (chloride, sulfate, and carbonate) that each contain a constant concentration of perchlorate.

4.1.1.1 Prepare the MCT standards described in section 2.2.7.

4.1.1.2 Measure and record the conductance of each of these prepared solutions on a conductivity meter.

4.1.1.3 Analyze each solution, recording the peak to area height ratio (A/H) and the quantified concentration of perchlorate.

4.1.1.4 Calculate the A/H ratio percent difference ($PD_{A/H}$) between the average A/H ratio for calibration standard #2 ($A/H_{CS\#2}$) and the average A/H ratio for each MCT standard (A/H_{MCT}) using the following equation:

$$PD_{A/H} = \frac{|A/H_{CS\#2} - A/H_{MCT}|}{A/H_{CS\#2}} * 100$$

4.1.1.5 The MCT is set at the conductivity of the highest MCT standard which yielded a $PD_{A/H}$ less than 20%.

4.1.1.6 Finally, confirm the reporting limit of perchlorate with a solution that has a conductivity within 10% of the MCT and a perchlorate concentration at or near that reporting limit. The recovered perchlorate concentration must be between 70-130% the true value. If the recovery falls outside of this criteria, then the MCT must be lowered by 10% and this procedure repeated.

4.1.1.7 The conductivity of all samples must be determined prior to analysis. If the conductivity is greater than the MCT, the sample must be diluted or pretreated.

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on the determination of method detection limits (LTL-1011). This SOP can be found in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration 1-5 times the anticipated method detection limit. A Student's t-test is then applied to these measured values to calculate the MDL.

4.2.2 MDL studies for the ion chromatograph must be determined at a minimum of every six months, with each new analyst, or whenever there is a significant change in system response. The current approximate MDL is 0.5 µg/L.

4.3 Determination of Retention Time Windows

4.3.1 Every six months, or any time there is a significant change in the system (such as analytical column replacement), the retention time windows need to be determined.

4.3.2 The RT window is determined by taking the retention times of three CCVs (mid range standards) on any one day, and computing the standard deviation. The standard deviation multiplied by three is the retention time window. This procedure is similar to that described in the Laucks SOP on Determination of Retention Time Windows (LTL-8000) except one run is used rather than a 72-hour period. This value is then entered into the AI-450 method. The retention time windows are stored in the method.

4.3.3 This data is generated on an Excel spreadsheet and is archived on the laboratory data base.

4.4 Initial Multi-Point Calibration

4.4.1 The reference method requires a calibration curve consisting of a minimum of five points if the curve extends over two orders of magnitude. LTL currently uses a six point curve. All calibration standards are diluted in sample vials at the instrument just prior to analysis as stated in the following table:

Standard Number	Volume of Standard	Volume DIW	Total Volume	Perchlorate concentration (µg/L)
1	50 µl of Cal #2	450 µl	500 µl	2.5
2	100 µl of Cal #2	400 µl	500 µl	5.0
3	500 µl of Cal #2	0 µl	500 µl	25.0
4	100 µl of Cal #1	400 µl	500 µl	80.0
5	250µl of Cal #1	250 µl	500 µl	200.0
6	500 µl of Cal #1	0 µl	500 µl	400.0

4.4.2 Samples whose responses exceed the high standard must be diluted.

4.4.3 A multi-point calibration of the system is not required with every analytical run.

4.4.3.1 If the previous calibration is verified by an ICV, the calibration is considered valid and the instrument does not require re-calibration.

4.4.3.2 A curve is not valid for a period of greater than six months.

4.4.4 Criteria

4.4.4.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The linear correlation coefficient (r) must be 0.995 or greater. All CCVs and sample concentrations must be computed using the regression equation.

4.4.5 Corrective action

4.4.5.1 If the criteria are not met the instrument must be recalibrated. No data may be reported for an analyte that does not have a valid calibration.

4.5 Instrument Performance Check

4.5.1 The MCT must be verified at the beginning of every analysis by analyzing an instrument performance check standard (IPC). The calculated $PD_{A/H}$ (see section 4.1.1.4 for calculations) of this solution must be less than 25% and the percent recovery of perchlorate must be between 80-120% of true value. Samples may not be analyzed and instrument maintenance must be performed or the MCT redetermined if the results fall outside this criteria. Also, the retention time of perchlorate must be within 80% of the original retention time of the column at installation. If the retention times have shifted past this point, then the column must be cleaned or replaced.

4.6 Initial Calibration Verification

4.6.1 At the start of each analytical run, analyze a standard from a source other than that from which the calibration material was obtained.

4.6.2 Criteria

4.6.2.1 The calculated concentration of the ICV for perchlorate must not to exceed ±25% of the true value.

4.6.3 Corrective action

4.6.3.1 If the ICV criteria are not met, no samples can be analyzed. Perform system maintenance and re-check the ICV. If the criteria still cannot be met, the system must be recalibrated. No data can be reported for an analyte that does not have a valid ICV.

4.7 Initial Calibration Blank

4.7.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed the 1/2 the reporting limit.

4.7.2 Corrective action

4.7.2.1 If the ICB contains target analyte levels above the 1/2 the reporting limit which is currently 4.0 µg/L the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis. No data can be reported for an analyte that does not have a valid ICB.

4.8 Blank Spike/LCS

4.8.1 Immediately after the analysis of the ICV and ICB, a blank spike (currently spiked with 50 µL of calibration standard #2) must be analyzed using the same solution used to calibrate the instrument and spike the matrix spikes. This sample is filtered and analyzed as a sample. If some samples are pretreated, then a separate blank spike that is also pretreated must be performed.

4.8.2 The blank spike recovery of perchlorate must be within 85-115%. If the recovery falls outside of this criteria, the source of the problem must be identified and resolved before continuing analysis.

4.9 Continuing Calibration Verification (CCV) and Blank (CCB)

4.9.1 A mid-range calibration standard is analyzed at a minimum of once every 10 sample injections. Immediately following the CCV, a blank solution is analyzed. In addition, at the end of the run, this standard should be run at a different level than preceding CCVs to verify that the curve has remained linear throughout the analysis. A CCB should follow this final CCV.

4.9.2 Criteria

4.9.2.1 The CCV must fall within ± 15% of the true value. The levels of target analytes in the CCB should not exceed the 1/2 the reporting limit of 4.0 µg/L.

4.9.3 Corrective action

4.9.3.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful ICV and a CCV which is in control or by preceding and following CCVs which are within limits.

4.9.3.2 If the CCB contains target analyte levels above the 1/2 the reporting limit of 4.0 µg/L, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following CCBs which are within limits.

4.10 Method Blanks

4.10.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time, or at least one blank every 20 samples, whichever is more frequent. Any analyte response above the reporting limit of 4.0 µg/L is reported. For the purpose of water analyses, the ICB, CCBs, and the method blank are the same. Method blank control limits are that contamination should not exceed the 1/2 the reporting limit for waters. Method blanks should not exceed the reporting limit of 100 µg/kg for soil leaches.

4.10.2 Corrective action

4.10.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interferences caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.11 Matrix Spike/Matrix Spike Duplicate

4.11.1 Project-specific criteria will determine whether MS/MSD or MS/Duplicate analysis is to be performed. Samples are spiked by adding 50 µl of calibration solution #1 to an aliquot of sample and diluting to 500 µl. The dilution factor of the sample and the spike are recorded in the dilution field of the batch schedule. The reference number for the spike is recorded in the sample name field of the batch schedule.

4.11.2 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the

minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\% \text{ recovery} = \frac{(SS - S) * 100}{SA}$$

where: SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.11.3 The recovery criteria are detailed in the Quality Control Database and will change from time to time. The current control limits are 80-120%.

4.11.4 Corrective action

4.11.4.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.12 Sample Duplicate

4.12.1 Criteria

4.12.1.1 At least one duplicate sample per 20 is required for Method 314.0. RPD values are calculated in the following manner:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where: S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.12.1.2 The RPD control limits are detailed in the Quality Control Database and will change from time to time. The current control limit is 15%.

4.12.2 Corrective action

4.12.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation Procedures

5.1 Initial Multi-Point Calibration

5.1.1 Creating a method

5.1.1.1 To start the Method Editor program double-click on the Method icon in the AI-450 MainMenu.

5.1.1.2 Method Settings:

System: Dionex DX-100
Number of Detectors: 1
Detector 1: Other-COND conductivity detector
1000mV=30.000uS
Run Time: 6-13 min. depending on retention time of sulfate peak
Plot Min.: -1
Plot Max: 30
Sampling Rate: 2 Hz
Timed Events: Init-Pump on
0.1:Offset on, Sampler on, Pump on, Begin Sampling on
Detector 1: Integration- Threshold-10.0, Width-10.0 sec, Reject-1000.0, Reference Peak area reject-2000.0
Data Events- void volume treatment for this peak at Fluoride retention time
Calibration- 4 levels, linear, calibration update- replace, standardization- external, Resp. for unknowns-area. The rest is set with the default settings.
Components- Enter each component that will be analyzed and their respective retention times, retention time windows. Calibrate by area. Reference component #1.

Enter amount of analyte at each calibration level.
Save new method as icMMDDYY.met where M=month, D=day, Y=year.

5.1.2 Creating a Calibration Curve Schedule

5.1.2.1 To start the Schedule Editor Program, double-click on the [Schedule] icon in the AI-450 MainMenu.

5.1.2.2 Enter sample name. For calibration purposes *autocal1r* is the name for the level 1 standard, *autocal2r* is the name for the level 2 standard, etc.

5.1.2.3 Enter the name of the method saved in 5.1.1.2

5.1.2.4 Enter a file name cMMDDYY, where M=month, D=day, Y=year. The program will number the filenames during analysis.

5.1.2.5 Save Schedule as cMMDDYY.sch., where M=month, D=day, Y=year.

5.1.3 Starting a run

5.1.3.1 Double-click on the [run] icon in the AI-450 MainMenu.

5.1.3.2 Load the schedule into the run program.

5.1.3.3 Load autosampler with cartridge containing the 4 levels of standards. Set autosampler to Run.

5.1.3.4 Set DX-100 to relay control.

5.1.3.5 Choose the run menu and start in the Run Program of AI-450.

5.2 Analysis tools and observations

5.2.1.1 If a discrete bottle number is referenced on the sample container, this information must be recorded in the sample name field of the batch schedule.

5.2.1.2 Samples that appear overly turbid or that have suspended or floating particles must be filtered through a 0.45 µm syringe filter prior to analysis. If this procedure is followed it should be documented.

5.2.1.3 Other anomalies should be discussed with the departmental supervisor in order to determine appropriate corrective action.

5.3 Sample Analysis

5.3.1 If samples are stored at 4±2°C, then samples should be brought up to room temperature prior to analysis.

5.3.2 Measure and record sample conductivity. If sample conductivity is less than the MCT, then the sample may be analyzed without pretreatment or dilution. If the sample conductivity is greater than the MCT, then samples must be pretreated or diluted prior to analysis.

5.3.3 Dilution

5.3.3.1 Calculate the dilution factor by dividing the measured sample conductance by the MCT and rounding up to the nearest whole number.

5.3.3.2 After dilution, measure and record the conductivity of the diluted sample to verify that it is below the MCT.

5.3.3.3 The sample reporting limit will change by an equivalent factor.

5.3.4 Pretreatment

5.3.4.1 Thoroughly rinse each filter cartridge with DIW to reduce the risk of contamination.

5.3.4.2 Rinse 3 mL of sample through the rinsed filter cartridges stacked in series (→Ba→Ag→H) at a flow of 1 mL/min. or less. This fraction is discarded.

5.3.4.3 Continue filtering at a rate of 1 mL/min until sufficient volume has been collected to measure the conductivity and analyze the sample.

5.3.4.4 Measure the conductivity of the pretreated sample to ensure that the conductivity is less than the MCT. If the sample conductance is below the MCT, then the sample may be analyzed. If the sample conductance is greater than the MCT, the flow rate through the filters is probably too fast and the sample pretreatment should be repeated with new cartridges.

5.3.4.5 If a batch contains pretreated samples, then a blank spike, method blank, and MS/MSD or MS/Dup must also be pretreated and analyzed.

5.4 Analysis sequence

Calibration standards (from low to high concentration)

IPC
ICV
ICB
Blank spike
Method blank
Pretreated blank spike (if necessary)
Pretreated method blank (if necessary)
10 or fewer injections
midrange standard (CCV)
DIW (CCB)
additional samples (not more than 10 injections)
additional CCVs and CCBs
...
...
must close with CCV and CCB

5.5 Instrumental Conditions

5.5.1 System Conditions

- Eluant flow is approximately 1.5 mL/min
- Injection volume is 500 µL.
- Temperature correction factor is 1.7.
- Detector range is 30 µS.
- Analysis time is approximately 15 minutes.
- Background conductivity is between 2-3 µS .

5.6 Analytical Operation

5.6.1 Analytical procedure for liquid matrixes after any necessary pretreatment.

5.6.1.1 A schedule is constructed for the sample set. This includes the appropriate dilutions for the given samples, the method name that corresponds to the curve that is in effect, and the filename where data for the sample will be saved. The file name extension is updated during analysis, therefore the analyst will not need to type in an extension.

5.6.1.2 The schedule is loaded into the run program of AI-450.

5.6.1.3 An appropriate number of 500 µL sample vials are placed into the auto sampler trays.

5.6.1.4 500 µL of standards, samples and QC events are placed into the sample vials.

5.6.1.5 Dilutions up to 50x can be done directly in the sample vials, greater dilutions will require an external dilution in a Solo cup or a combination of external and sample cup dilutions.

5.6.1.6 Sample vials are capped with filter caps and placed in the auto sampler.

5.6.1.7 The "Control" button on the DX-100 is set on relay and the autosampler is set on local control and run.

5.6.1.8 Choose the run menu and start in the Run Program of AI-450.

5.6.2 Analytical procedure for soil or sludge matrixes.

5.6.2.1 The soil/sludge is leached with DIW by transferring 10 g to a tared, 250 mL digest bottle, adding 100 mL DIW and placing on the shaker for 15 minutes or more at a shaking rate sufficient to suspend the majority of the solids. The solids are then allowed to settle and an aliquot of the supernatant is filtered through a 0.45 µm syringe filter prior to use. The leaching and filtration of the sample must be recorded in the pretreatment field of the batch summary and a prep sheet completed.

5.6.2.2 A 100 mL aliquot of DIW is shaken in a 250 mL digest bottle and used as a method blank.

5.6.2.3 The resulting supernatant is treated as a water sample and is pretreated if necessary.

5.6.2.4 A total solids determination is made on all soil/sludge samples and the results are reported on a dry weight basis.

5.7 Compound Quantification

5.7.1 The data acquisition system generates a report with the chromatogram of the sample, the date analyzed, the sample identification, the method used to quantify the sample, the sample dilution, and the calculated concentration of the sample corrected for dilution.

5.7.2 For soil/sludge samples, the mg/L integrator value is converted to mg/kg DB by the following equation:

Soil: $$Cs = \frac{Ci * Vs * 100}{Ws\ TS}$$

Cs = Sample concentration (mg/kg DB)
Ci = Concentration in sample injected (mg/L)
Vs = Volume of extract (mL)
Ws = Weight of sample extracted (gm)
TS = Total solids (as fraction)

6. Reports

6.1 Sample Results and Quality Control Reports

6.1.1 Data packages shall consist of the sample results summary, the chromatograms for samples in the workorder, and a copy of the QC summary report form. These include the method blank analysis, matrix spike results, duplicate results, and the Standard Reference Material (SRM) reports. A copy of the QC summary report form must be included in order to indicate that the QC was evaluated for analytical consistency.

6.2 Data Qualifying Flags

6.2.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

Appendix I

Method 314.0 QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	A minimum five point curve for curves that span two orders of magnitude that brackets the sample concentrations.	A six point curve that brackets the sample concentrations Corr coef r≥ 0.995	As instrument performance dictates, or when any change is made in the analytical system.	Fix the problem and recalibrate.	In the raw data package for all validatable packages and referenced in all other data.
Instrument Performance Check (IPC)	Conductance at ± 10% of the MCT; $PD_{A/H}$ less than 25%; Perchlorate recovery at between 80-120%. Perchlorate retention time greater than 80% the original RT.	Conductance at ± 10% of the MCT; $PD_{A/H}$ less than 25%; Perchlorate recovery at between 80-120%. Perchlorate retention time greater than 80% the original RT.	Prior to analysis.	Repeat the MCT study and reanalyze the IPC; Clean analytical column.	In the raw data package for all validatable packages and referenced in all other data.
Initial Calibration Verification	Not to exceed ±25% of the true value.	Not to exceed ±25% of the true value.	At the beginning of the analysis.	Reanalyze, if reanalysis fails recalibrate and or perform instrument maintenance. No data can be reported if ICV fails.	In the raw data package, and in the quality control data base.
Initial Calibration Blank	Value not to exceed the RL.	Value not to exceed the 1/2 the reporting limit	Immediately following the ICV.	Reanalyze. No data can be reported if ICB fails.	In the raw data package, and in the quality control data base.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Continuing Calibration Verification	Not to exceed ±15% of the true value.	Not to exceed ±15% of the true value.	At a minimum, after every 10 injections and as the final injection.	Samples which are not bracketed by in control CCVs must be reanalyzed.	In the raw data package.
Continuing Calibration Blank	Value not to exceed the RL.	Value not to exceed the 1/2 the reporting limit.	Immediately following a CCVs.	Samples which are not bracketed by in control CCBs must be reanalyzed.	In the raw data package.
Blank Spike/LCS	Spike a blank with the calibration standard at no greater than 10 times the highest perchlorate level in the sample batch; recovery at +/- 15% of true value.	Spike a blank with the calibration standard at no greater than 10 times the highest perchlorate level in the sample batch; recovery at +/- 15% of true value.	Run at a minimum of 1 per 20 samples. A pretreated BS must be run with samples that are pretreated.	Source of the problem identified and fixed prior to analyzing samples.	Run at a minimum of 1 per 20 samples. A pretreated MB must be run with samples that are pretreated.
Method Blank	Value not to exceed the RL.	Not to exceed the 1/2 the reporting limit for waters and the reporting limit for soils.	Run at a minimum of 1 per 20 samples. A pretreated MB must be run with samples that are pretreated.	If sample concentration exceed 10x the contamination level, no action required. Samples with contaminated Method Blanks must be re-extracted.	In the raw data package, and in the quality control database.
Matrix Spike /Matrix Spike Duplicate Recovery	Not to exceed ±20% of the true value. Once every 20 samples.	Not to exceed ±20% of the true value. Once every 20 samples.	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control database, and in raw data package.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Duplicate/ Relative % Difference (RPD)	Not to exceed 15%. Once every 20 samples	Not to exceed 15%. Once every 20 samples	Once every 20 samples, or per client request, whichever is more frequent.	Review data, consult QC officer, some conditions may require reanalysis.	Narrative comment in report in quality control database, and in raw data package.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-9202

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APPENDIX I **11**

QC SUMMARY TABLE 11

APPENDIX II **13**

FLOW CHART 14

1. Introduction and Scope

1.1 Method Description

1.1.1 Scope and Application

1.1.1.1 This method is applicable to drinking, surface, and saline waters, domestic and industrial wastes. The practical range of the determination is 4 mg/l to 20,000 mg/l. The detection limit, however, is generally reported as 2 mg/l unless a sample volume greater than 100 mls is used.

1.1.1.2 A well-mixed sample is filtered through a standard glass fiber filter. The filter and residue are dried to constant weight at 103°C-105°C.

1.1.1.3 The filtrate from this method may be used for Total Dissolved Solids (Filterable Residue).

1.1.1.4 If volatile suspended solids are to be determined, the filter and residue are then ignited at 550°C and the loss on ignition determined.

1.1.2 Interferences

1.1.2.1 Samples high in dissolved solids (filterable residue), such as saline waters, brines and some wastes, may be subject to positive interferences due to soluble material which has not been adequately washed from the filter. Care must be taken to ensure that an appropriate filtering apparatus has been selected and the filter adequately washed in order to minimize this possibility.

1.1.2.2 Samples which are very high in suspended material or which have certain particle sizes may plug the filter causing difficulty in filtering. It may be necessary to reduce sample size in order to reduce this tendency.

1.1.2.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Sample Collection, Sample Storage, Holding Times

1.2.1.1 No preservation of the sample is necessary. Refrigeration or icing to 4°C, to minimize microbiological decomposition of solids, is required. Analysis should begin as soon as possible and should in no case exceed 7 days from the date of collection. All exceptions, whether due to laboratory or client cause, must be recorded on a Holding Time Violation Report (HTVR).

1.3 Definition of Terms

1.3.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

MDL - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater zero at a known level of confidence. MDLs are empirically determined by Laucks, although there is no known way to determine the MDL for the TSS or TVSS analyses.

SRM or LCS - Standard Reference Material or Laboratory Control Sample. This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials. This type of sample is rarely analyzed at Laucks for TSS and there is no known source of TVSS material.

Sequence - A set of samples analyzed in a chronologically continuous group.

Total Suspended Solids (TSS) (nonfilterable residue)- Those solids which are retained on a glass fiber filter of the appropriate retentive ability and dried to constant weight at 103-105C.

Total Volatile Suspended Solids (TVSS) - Suspended solids which are volatile (burn off) at a temperature of 550°C.

2. Equipment List

Glass fiber filter discs, 4.7 cm without organic binder, Gelman A/E or equivalent. Laucks currently uses Pro Weigh 47 mm glass fiber filters from Environmental Express which are designed for TSS analysis. They are pre-washed and pre-weighed and require no additional preparation.

Membrane filter funnel with the capability of adequately supporting 4.7 cm. filters

Suction flask

Drying oven, set at 103°C-105°C.

Muffle furnace set at 550°C ± 50°C

Desiccator charged with active silica gel desiccant

Analytical balance, capable of weighing to 0.1 mg.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Routine precautions include an awareness of elevated temperatures of both the oven and any samples which have recently been removed from the drying oven or especially the muffle furnace. The temperatures involved in this analysis can cause severe burns if adequate care is not taken.

3.2 Waste Disposal

3.2.1 No waste should be generated from this procedure. Sample residues may be washed out and discarded down the sink.

4. Quality Control

4.1 Method Blanks

4.1.1 Criteria:

4.1.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples which ever is more frequent. The method blank is prepared by pouring the same amount of deionized water through a filter as one would a typical sample (generally 100 mls.). Any TSS above the detection limit is reported. Method blank control limits are such that contamination should not exceed twice the detection limit. If 100 ml samples are used, Laucks generally considers the detection limit to be 2 mg/L with method blank control limits of 4 mg/L.

4.1.1.2 In making a determination of whether or not the analysis is in control, the analyst should actually be normalizing the blank to whatever volume was used for any sample. It is assumed that any blank is due to washings from the filter and apparatus and is actually independent of sample volume. In other words, the control limit for this analysis is actually 0.4 mg. If blank contamination exceeds that value, the TSS (or TVSS) of samples must be ten times that weight or corrective action should be taken.

4.1.1.3 If TVSS is being determined, the filters are processed in the same manner only are also heated to 550°C. Otherwise, the detection limits, criteria, and corrective actions are the same as for TSS.

4.1.2 Corrective action

4.1.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if TSS or TVSS were found in the blank but not in any of the associated samples, then the sample group may not require re-analysis. In addition, if sample levels exceed 10 times the blank, the level of contamination may be considered insignificant. In any case, if re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.2 SRM or LCS

4.2.1 Criteria

4.2.1.1 Analysis of a reference material is not normally required for TSS analysis and no known material is available for TVSS. An SRM/LCS analysis will generally be analyzed only if specifically required for a project. If not otherwise specified in that contract, it would typically

be analyzed at a frequency of once per 20 samples. Vendor specified control limits would be used for any such material.

4.2.2 Corrective Action

4.2.2.1 Re-analysis of all associated samples may be required if this sample exceeds it's limits. The QC Officer should be consulted for any other corrective actions and all instances of out-of-control events and any actions taken must be documented in the QC narrative of the report.

4.3 Sample Duplicate

4.3.1 Criteria
At least one duplicate sample per 10 samples is required. RPD values are calculated as follows:

$$RPD = \frac{|S1 - S2| * 100}{(S1 + S2)/2}$$

where
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analysis

4.3.1.1 The RPD control limits are detailed in the Quality Control Database (QC_DB) and will change from time to time. For samples with values which are less than 5 times the detection limit, the control limit is equal to 5 times the detection limit. For values greater than 5 times the detection limit, the control limit is a calculated percent RPD.

4.3.2 Corrective action

4.3.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. If integrity of reported sample values is in doubt, re-analysis of all associated samples may be called for. Corrective actions should be discussed with the Quality Control Officer.

5. Operation procedures

5.1 Sample Analysis

5.1.1 Analysis sequence

Method Blank
SRM or LCS (if required)
up to 20 samples plus duplicates

5.1.2 Analytical Operation

5.1.2.1 Drying, ashing or cooling times in this section are those typically used in the laboratory and are not specifically required by the method. The method requires a minimum of 1 hour drying or ashing times and analysis to constant weight. This may be achieved through either shorter or longer time periods than those specified in this SOP. Experience has also shown that the beakers or other weighed containers must be cooled to room temperature for the weights to be stable and accurate. As long as proper and complete cooling has been allowed to occur in an environment where the dried or ashed sample cannot re-absorb water or be otherwise contaminated, these times are not specified in the published method and are thus also flexible.

5.1.2.2 Preparation of glass fiber filter disc if Environmental Express Pro Weigh filters are being used: Remove the filter from it's aluminum weighing dish and place the disc on the membrane filter apparatus with the wrinkled side up. While vacuum is applied, rinse the disc with a small amount of deionized water in order to seat the disk in the filter device.

5.1.2.3 Preparation of glass fiber filter disc if pre-washed and pre-weighed filters are NOT being used: Place the glass fiber filter on the membrane filter apparatus (if 4.7 mm filters are being used) or insert into bottom of a suitable Gooch crucible (if 2.4 cm filters are being used) with the wrinkled surface up. While vacuum is applied, wash the disc with three successive 20 ml volumes of distilled water. Remove all traces of water by continuing to apply vacuum after water has passed through. Remove filter from membrane filter apparatus (if 4.7 mm filter) or both crucible and filter if Gooch crucible is used, and dry in an oven at 103°C-105°C for one hour or at 550°C ± 50°C if TVSS is to be determined. Remove to desiccator and store until needed. Repeat the drying cycle until a constant weight is obtained (weight loss is less than 0.5 mg). Weigh immediately before use. After weighing, handle the filter or crucible/filter with forceps or tongs only. As in the procedure for the pre-weighed filters above, wet the filter slightly before use in order to properly seat it in the filtration apparatus.

5.1.2.4 Assemble the filtering apparatus and begin suction, taking care to have wet the filters as described in either procedure above. Shake the sample vigorously and rapidly transfer 100 ml to the funnel by means of a 100 ml graduated cylinder. If this volume takes longer than 5-10

minutes to pass through the filter, sample volume must be reduced such that the filtration time will not be exceeded. If that volume is less than or equal to 10 mls, it should be dispensed with a 10 ml Mohr (graduated glass) pipet which has a wide enough tip opening so as not to inhibit the passing of solid pieces of material.

5.1.2.5 Filter the sample through the glass fiber filter, rinse the graduate and filter with three successive 10 ml portions of deionized water, allowing the rinsate to pass completely through the filter between washings, and continue to apply vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

5.1.2.6 If the sample contains large pieces of material which make it difficult or impossible to achieve a representative, homogeneous sample, it may be necessary to thoroughly mix the sample in a blender prior to filtration. If this is done, however, care should be taken to assure that air bubbles aren't entrained in the measured sample volume to the extent that it could affect the actual volume dispensed. In other words, let the bubbles and subsequent foaming subside before dispensing the sample.

5.1.2.7 Carefully remove the filter from the filtration device, taking care not to leave pieces of filter on the support apparatus, and place it in the aluminum weighing dish appropriate to that filter and sample.

5.1.2.8 Dry the filter overnight at 103°C-105°C.

5.1.2.9 Remove the filter and aluminum dish from the oven and place in a desiccator to cool. The samples MUST be cooled prior to weighing or the apparent weight will be affected.

5.1.2.10 Repeat the cycle of drying, desiccating, cooling and weighing until the filter/dish attain a constant weight, changing by no more than 0.5 mg.

5.1.2.11 Record the final weight for calculation of TSS.

5.1.2.12 If TVSS is to be determined, repeat step 5.1.2.9 using a muffle furnace pre-heated to 550°C ± 50°C for 90 minutes. Record the final weight for calculation of TVSS.

5.2 Quantification

5.2.1 Residue concentrations are calculated using the following equations:

$$TSS = \frac{(W_f - tare) \times 1000}{V_i}$$

$$TVSS = \frac{W_f - W_a \times 1000}{V_i}$$

where:

V_i = volume of sample used in mls
W_f = weight of dried residue & filter
W_a = weight of ignited residue (after ignition @ 550°C) & filter in mg
tare = tare weight (weight in mg of filter before filtration of sample)

5.2.2 TSS and TVSS are generally reported on a mg/L as received basis.

6. Reports

6.1 Data Packet Organization

6.1.1 The data package for this analysis consists of the data sheet and a quality control database (QC_DB) report form.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the lab data base using the QC_DB program. Printouts of all data entered need not be included in the package. However, all must be referenced on the report form. This includes the blank and duplicate results and any other QC which might have been analyzed by special request (such as an SRM/LCS).

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

CODE	Definition
U	: The analyte of interest was not detected, to the limit of detection indicated.

Appendix I

QC Summary Table

Laucks Testing Laboratories

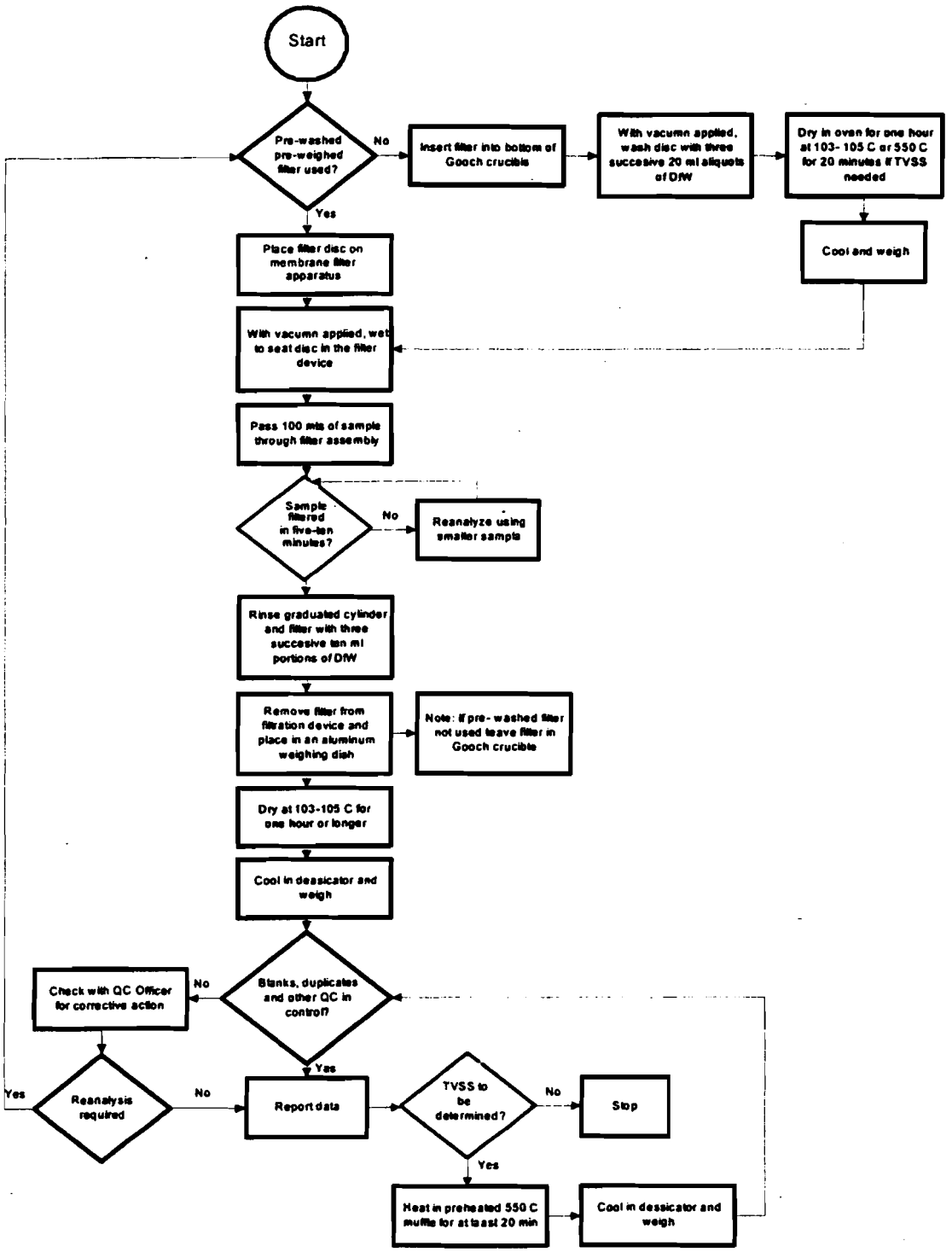
Method EPA 160.2 / SM 2540D & E QC Requirements and Corrective Actions

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Method Blank	None	<4 mg/L or no more than twice the detection limit	5% frequency (1 per 20 samples)	Re-analyze all samples <10x the actual weight of the solids. Consult QC Officer for any other actions	QC_DB report form with appropriate commentary
Duplicate % Difference	None	See current control limits catalog or QC_DB database	10% frequency (1 per 10 samples)	Discuss with QC Officer. If impact appears serious, may need to re-analyze samples	QC_DB report form with appropriate commentary
Standard Reference Material (SRM) Recovery	None	Within vendor supplied limits or 90%-110% recovery	If required, at frequency specified, or 5% if not specified	Discuss with QC Officer. If impact appears serious, may need to re-analyze samples	QC_DB report form with appropriate commentary

Appendix II

Flow Chart

Total Suspended Solids (TSS) and Total Volatile Suspended Solids (TVSS)
EPA 160.2 and/or SM 2540D&E



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NON-CONFORMANCE				
TLI SOP No.	2.04	Version:	6	Date Revised: July 27, 2001

Author: Jennifer Hass Effective Date: 1 August 2001

Authorization: Donald Harvan Date Authorized: 30 July 2001
Management

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I. **DEFINITION:** A Non-Conformance is the failure to comply with one or more criteria established for the acceptance of data.

II. **SCOPE AND APPLICATION:** The purpose of this SOP is to describe the decision and documentation process applied to non-conformance's or analytical elements which fail to meet the applicable acceptance criteria. This SOP applies to all analytical data and associated data reports, and must be followed by all members of the TLI staff.

III. **RESPONSIBILITY:** The staff member who identifies a non-conformance issue is responsible for evaluating the failure condition and initiating the applicable course of action.

IV. **PROCEDURE:**

A. When analytical data do not satisfy the acceptance criteria, the staff member evaluates the extent of the failure using the Allowed Exceptions section of this SOP as a guideline.

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B. Allowed Exceptions

1. All Methods

Issue	Allowed Exception
Blank Contamination	1. if blank contamination is less than 5% of that analyte concentration in the client samples 2. if client samples have ND for that analyte 3. if client samples have "B" Flag and "J" Flag for that analyte
Instrument Spike	if it does not affect quantitation of analyte or put the standard out of ratio
MS/MSD analyte recoveries and relative percent differences	if it does not look like an obvious sample preparation error and the native client sample has high levels of analytes such that the amount of spike is small relative to the native amount
Quantitative Interference or Interference	if only in client samples and it appears to be a matrix problem
Ratio Out	for client samples only if due to high levels of analytes or obvious interference
Saturation	all conditions for only client samples, but project scientist must be notified

2. Method 8290 and Method 23

Issue	Allowed Exception
LCS/LCSD analyte recoveries	up to two analytes may have % recoveries as high as 145% and as low as 60% as long as associated relative percent differences meet criteria
LCS/LCSD relative percent differences	up to two relative percent differences may be 20-35% as long as the associated percent recoveries meet criteria
Labeled Standard Recoveries	internal standard recoveries may be as low as 25% if signal to noise is 10:1 for associated standard

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3. Method 8280

Issue	Allowed Exception
LCS/LCSD analyte recoveries	up to two analytes may have % recoveries as high as 165% and as low as 40% as long as associated relative percent differences meet criteria
LCS/LCSD relative percent differences	Up to two relative percent differences may be 50-65% as long as the associated percent recoveries meet criteria
Labeled Standard Recoveries	internal standard recoveries may be as low as 25% if signal to noise is 10:1 for associated standard

4. Method 1613

Issue	Allowed Exception
Blank Contamination	All blank contamination below the target detection limit is allowed by the method
Standard recovery limits for tetra-octa analyses	1. 13C12-2378-TCDD: 25%-164% 2. 13C12-2378-TCDF: 24%-169% 3. 37Cl4-2378-TCDD: 35%-197%

5. High resolution PCB analysis

Issue	Allowed Exception
Blank contamination	if blank contamination is less than 10% of that analyte concentration in the client samples
Low standard recoveries	Mono, di, and tri standards do not have recovery limits. However, if the recovery is less than 10%, an NCR should be created and the project scientist must approve it.

6. Method 680

Issue	Allowed Exception
Low standard recoveries	Mono, di, and tri standards do not have recovery limits. However, if the recovery is less than 10%, an NCR should be created and the project scientist must approve it.

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C. A project with a non-conformance which is not addressed in the Allowed Exceptions section of this SOP must have a Non-Conformance Report(NCR) initiated. The special instructions for an individual project override the Allowed Exceptions section of this SOP. Projects for which NCRs are initiated must be taken to the project scientist for approval prior to shipment. All non-conformances must be addressed in the case narrative, regardless of whether an NCR was created.

D. A completed NCR shall include the following:

1. Initials of the persons identifying the non-conformance and creating the non-conformance report;
2. Date the non-conformance is identified;
3. Identification of the affected project data (including identification of any associated batched projects);
4. A description of the failure or non-conformance;
5. The initiator's plan for resolution of the non-conformance
6. Project scientist is responsible for documenting a description of the planned actions, action associated with the investigation, and resolution of the non-conformance; and
7. Project scientist is responsible for documenting conclusions regarding the impact of the non-conformance on other project data.

E. Data in the NCRs shall be documented as required in the SOP for recording raw data.

F. Completed NCRs are reviewed and accepted by a Project Scientist. The Project Scientist evaluates the proposed actions and the potential impact on other data.

G. Technical guidance is sought from the Area Manager or appropriate member of senior management until an adequate solution has been defined for the non-conformance.

H. Project Scientist defines the planned action for resolving the non-conformance and documents resolution in MILES. Verbal acceptance by Project Scientist may be given after full discussion of the NCR issues. In such cases, the person on-site must document the verbal acceptance.

I. After resolution of NCR, a copy of the NCR and any supporting data not already in the project file are placed in the project file.

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J. Data associated with an NCR cannot be reported to the client until the NCR has been accepted. Any NCR issues and their affect on the data must be reported in the case narrative.

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SAMPLE RECEIPT, IDENTIFICATION, HANDLING AND STORAGE		
TLI SOP No. 5.01	Version: 10	Date Revised: August 13, 2001

Author: Anzor Gatchetchiladze Effective Date: 08-27-2001

Authorization: Donald Harvan Date Authorized: 20 Aug 2001
Management

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I. **PURPOSE:** To describe the standard operating procedures (SOPs) to be followed to ensure sample security and integrity from the time of receipt to the time of final disposal. This includes procedures for sample inspection, log in, storage, and disposal.

II. **SCOPE:** The procedures described in this SOP are applicable to all samples that are received by TLI's Environmental and Pharmaceutical Divisions.

III. **RESPONSIBILITY:** The primary responsibility for sample security and integrity belongs to the Sample Management Group or designated alternate(s), who initiates the procedures described in this SOP. However it is the responsibility of all personnel at TLI, who handle samples, to ensure the security and integrity of the samples under their control and to document on a COC form the movement of those samples.

IV. **SAFETY CONSIDERATION:** Incoming samples may contain harmful substances. Gloves, lab coat, and safety glasses must always be worn when handling samples. Some situations may require wearing a respirator. These situations include the receipt of leaking or highly volatile samples, or certain samples for which specific hazards are known prior to receipt. For samples of specific known hazards, refer to the appropriate appendix in this SOP for additional handling instructions. See Appendix A for handling samples contaminated with the hantavirus, and Appendix B for handling human blood and other potentially infectious material (OPIM). For additional safety information, consult the Safety and Health Officer or the "Safety and Health Manual".

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V. PROCEDURES:

NOTE: Special instructions from study specific protocols or EPA methodology supersede this SOP.

A. SAMPLE RECEIPT:

1. At the time a sample shipment is received, the receiving individual will document, the time and date received, who received it, and the general condition of the package(s). For Pharmaceutical samples Shipping Container Internal Custody Form must be filled out at this time.

   When the customer delivers the samples, also document the name of the individual making the delivery.

2. Open shipping container and carefully take all the samples out of the container. Take temperature of the samples (see paragraph B.3 of this SOP). Immediately count all the samples in the shipment and document on the Shipping Container Internal Custody Form (applies only to pharmaceutical samples). Examine all the documentation arrived with the shipment such as the Chain-of-Custody Report, Specimen Shipment Verification Form, Sample Traffic Report Form, Shipping Container Internal Custody Form, etc. Verify and confirm that number of actual samples in the shipment matches the number of samples indicated in the paperwork.

   NOTE: If any discrepancy is found at this point between the actual sample count and the shipment paperwork, stop the log-in process and notify the appropriate Project Scientist and the Sample Management Coordinator immediately. Do not proceed until clear instructions are obtained from the Project Scientist.

   a) If the external documentation indicates that the samples could potentially be contaminated with the hantavirus (i.e. deer mice, squirrels, cotton mice, field mice, etc.), refer to Appendix A for specific instructions.

      Notify the appropriate Project Scientist and Sample Management Coordinator immediately. It is the responsibility of the Project Scientist to contact the client so tracing procedures can be implemented.

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NOTE: At this point, if samples cannot be processed immediately, place the shipping container(s) in the appropriate cold storage location (See section D). It is not necessary to place frozen samples in the freezer if there is sufficient dry ice in the shipping container to keep the samples frozen until full inspection and log in can be performed. Document when and where the shipment was stored.

3. Take the shipment to the designated sample receiving area to be inspected and logged in. If the receiving individual does not perform the inspection/log in or the sample shipment was stored prior to inspection, document when and by whom the inspection/log in was performed.

   NOTE: All the samples must be logged-in within 24 hours of receipt unless instructed otherwise by Sample Management Coordinator or a Project Scientist.

4. Create a project number for each sample or group of samples received using the TLI's Management Information for the Lab Environment System (MILES). Project numbers are assigned sequentially by MILES. For samples of specific known hazards, follow the handling instructions detailed in the appropriate appendix for sample inspection and log in.

   NOTE: Only the Sample Custodian or a designated alternate may open sample shipping containers unless permission obtained from the Sample Management Coordinator.

B. SHIPMENT INSPECTION: The inspection of each shipment is documented in MILES, referencing the appropriate project number on each shipping document. Items to be documented include:

1. Shipping Container(s)

   a) Presence/absence of custody seals

   b) Condition of custody seal - intact/broken

   c) Description of shipping container (e.g. ice chest, Styrofoam cooler, box) and its general condition

   d) Carrier and airbill number(s)

2. Shipping Documents

   a) Presence/absence of client COC.

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b) Presence/absence of Sample Management Office (SMO) Forms (for EPA samples only).

c) Presence/absence of airbills and/or bills of lading.

3. Status of Preservation/Temperature Conditions

a) Presence/absence of coolant.

Is there frozen coolant present? Is there evidence that coolant was present but thawed en route? Is there no evidence of coolant ever having been present (i.e. shipped at ambient temperature)? Document what is observed.

b) For all samples received with some evidence of coolant (other than dry ice) and for samples to be analyzed by methods specifying preservation at 4 $\pm$ 2°C measure and record the temperature. For samples received on dry ice record on the Chain of Custody: "Dry Ice". No temperature should be recorded for these samples.

(1) If in doubt about preservation requirements/evidence of coolant, measure and record the temperature.

(2) Measure the temperature of the single randomly selected sample using a calibrated Infrared (IR) thermometer gun. NOTE: See Table 1 for common Emissivity values for materials.

(3) If the IR thermometer gun is unavailable for any reason, place an in-house temperature blank (a scintillation vial of deionized water which has been stored at approximately 4°C) in the shipping container (close the container) for at least one half hour and measure the temperature with a calibrated Celsius thermometer.

4. If the samples are not received under the specified temperature conditions, proceed with inspection/log in and notify the Project Scientist/Principal Investigator immediately to obtain further handling instructions from the client. Order Acknowledgement specifying sample conditions at the time of the sample receipt will be faxed to the client as soon as sample log-in/initiation process is complete.

5. Observe and document conditions of the sample containers:

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a) Presence/absence of sample custody seals.

b) If wild rodent carcasses are present but not indicated on external documentation, immediately close and secure the sample container and notify the Health and Safety Officer (HSO).

c) Condition of the samples (intact, broken, leaking, etc.) and action taken, if applicable.

(1) Photograph unfavorable sample shipments and file photos along with the copies of client COC in "Damaged Sample Logbook" maintained by Sample Management Group.

(2) Salvage leaking or broken sample(s), when doing so will not increase the likelihood of further sample loss or contamination as follows:

(a) If a leaking sample is discovered, it may suffice to simply tighten the lid. Otherwise, treat it like a broken sample (Note loose lid on the COC).

(b) If a bottle containing a solid sample is cracked, but remains intact, wrap the bottle in aluminum foil and place the container in a plastic bag with a "zipper" closure.

(c) Isolate each broken sample into an appropriate new container, which is labeled with the client sample ID.

(i) Wrap fish and paper in clean foil, with the dull side towards the sample.

(ii) If a tube containing a biohazard sample is cracked, but the sample is still frozen and intact, place the tube in a clean beaker. Allow the sample to thaw, then transfer to a new labeled test tube. This procedure must be performed by qualified Chemist.

(3) Presence/absence of Contract Laboratory Program (CLP) tags and numbers. Tags are normally attached around the neck of the bottle they apply to. Document

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any discrepancies between the number on the tag and the client paperwork. CLP sample tags will be removed from the samples and shipped with the data package.

(4) Notify the appropriate Project Scientist and Sample Management Coordinator immediately of any damaged or unidentifiable samples. It is the responsibility of the Project Scientist to contact the client to obtain further instructions on how to proceed with the affected sample(s) and verify any known or suspected hazards associated with the affected sample.

(5) The Sample Management Group will properly dispose of samples that cannot be recovered, after the Project Scientist's authorization.

6. Compare client COC, if present, with sample identification. Document any discrepancies, such as missing samples, samples present but not listed on the client COC, missing information or differences in information contained on the sample label/tag and the client COC. Notify the Project Scientist immediately of any discrepancies listed above. It is the responsibility of the Project Scientist to determine if the discrepancies require client's attention.

7. Return non-disposable shipment containers and coolant packs, which belong to the client, to the return address found on the airbill or client COC.

C. SAMPLE LOG IN AND IDENTIFICATION:

1. Every incoming sample is logged in into MILES "virtual logbook" which then creates TLI project number and TLI sample IDs for every sample or group of samples. From this point on every logged in sample can be tracked throughout the entire process until disposed or shipped back to the client.

2. TLI IDs assigned to samples are unique ID that represent:

a) "Virtual Logbook" Number - Page Number - Sample Sequence Number. For example: sample logged into Virtual

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Logbook # 258, on page # 16, sample sequence # 2 will translate into TLI sample ID: 258-16-2

b) If a single sample is submitted in more than one container, each container is uniquely identified by appending sequential letters (A through Z) to the Sample Sequence Number. Ex: 258-16-2A, 258-16-2B, etc.

3. For each sample, record the client sample identifier as stated on the sample label and client COC. Also, record sample matrix, and the initial sample storage location. If samples will be scanned with Bar-code scanner refer to the appropriate SOP for using, recording, and transferring data obtained with bar-code scanner.

4. Securely label each sample with the TLI project number and both, TLI and client sample IDs.

5. Generate the TLI COC, sign and date it. If applicable, generate any other necessary paperwork, such as: Log in Form, Extract COC and Project Communication forms (usually required for Pharmaceutical samples only). Sign and date the paperwork.

6. The nature of the project determines the exact storage location of all original shipping and log in documents for the project.

   a) In all cases, documentation will be identified by project number and will be maintained in such a manner as to prevent destruction or loss (i.e. file cabinet, study binder, project file, etc.).

   b) Any copies of these documents will be clearly labeled as such and will be initialed and dated.
   NOTE: During the entire log in process, including sample inspection and labeling, all samples must be maintained at a temperature that insures sample integrity.

D. SAMPLE STORAGE:

1. Place samples in boxes, crates or coolers, as appropriate for the sample matrix. Label storage container with TLI project number in such a way as to avoid breakage and cross contamination.

   a) Never store samples together with chemical standards in the same refrigerator/freezer.

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b) For Pharmaceutical projects, note the location of the samples on the appropriate inventory list.

c) Store Pharmaceutical samples in freezers designated by Project Scientist on the Sample Log in And Initiation Form.

2. Actual storage requirements are dependent upon the matrix, the analysis requested, and client's protocols or specifications. Storage temperature can vary from Room Temperature to 4°C, - 20°C or - 70°C.

E. SAMPLE TRACKING:

1. All personnel handling samples (extracts) at any time before, during or after analysis, will document the movement of the sample on the original TLI COC form. Original TLI COC forms are stored in the storage containers along with the samples. For Pharmaceutical samples original TLI COC forms are placed in the binders located outside of every freezer door. Copies of COC forms (Log-in Forms for Pharmaceutical projects) are placed in the project folders. Documentation includes:

   a) The identification of the sample being moved.

   b) The initials of the person who removed and returned the sample from and to the storage location.

   c) Date when the sample was removed and returned from and to storage. If returned to a different location - the new storage location.

2. In addition, the sample's whereabouts within the analytical process will be tracked via MILES system.

   NOTE: Copies of COC forms must be clearly labeled as such and be signed and dated. Entries and updates must be made on the original document.

F. SAMPLE DISPOSAL OR RETURN:

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1. Sample disposal will be performed in compliance with the state and federal regulations. Unless instructed otherwise by the client, samples will be disposed as follows:

   a) All samples will remain in the appropriate storage location throughout the analysis process.

   b) Environmental samples will be disposed 30 days after the complete data package ships to the client. It is a client's responsibility to arrange sample storage for longer than 30 days if necessary. Sample storage fees may apply in this case.

   c) Pharmaceutical samples will be disposed 90 days after the final report is issued. During this 90 days client will be contacted by Project Scientist and further storage, disposal or sample return will be arranged. Sample storage fees may apply if client chooses to keep samples at Triangle Labs storage locations longer than 90 days.

   d) After samples are disposed the original TLI COC form will be updated and then archived.

   e) In addition the MILES system will be updated as soon as the original COC is updated. For details, please see Sample Disposal SOP.

2. Sample Return

   a) Obtain client's authorization and packing/shipping instructions before returning any samples.

   b) Sign and date original client's COC and/or packing list for all samples being returned. Original will accompany the shipment and a copy will be retained for the project (study) file at TLI.

   c) Pack samples to prevent breakage and contamination.

   d) Pack samples/extracts on ice, dry ice or without coolant at all, as requested by the client.

   e) Document sample return on TLI COC form and update MILES.

If this SOP can not be followed notify your supervisor immediately.

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APPENDIX A

PROCEDURE FOR HANDLING RODENT SAMPLES POTENTIALLY CONTAMINATED WITH HANTAVIRUS

NOTE: CONTACT THE SAFETY/HEALTH OFFICER IMMEDIATELY. DO NOT OPEN THE SHIPPING CONTAINER UNTIL AUTHORIZED BY HIM/HER.

I. **SCOPE AND APPLICATION:** The purpose of this document is to outline the supplies and specific procedures for the safe handling of rodent samples (i.e. deer mice, squirrels, cotton mice, field mice, etc.) that are potentially contaminated with hantaviruses of significance to human beings.

II. **SUPPLIES REQUIRED:**

- Two half-mask respirators with organic vapor/acid gas/HEPA cartridges.
- Two non-vented splash goggles.
- Two tyvek coveralls.
- Two plastic splash aprons.
- Four pairs latex gloves.
- Commercially available chlorinated bleach diluted 1:10 with water. (10% bleach disinfectant) in a spray bottle.

III. **PROCEDURE:**

A. The samples will be delivered directly to Triangle Laboratories. Shipping containers will remain closed until they are ready for processing. Freezing of the entire unopened shipping containers is allowed if they arrive late in the day for processing during the subsequent six hours.

B. All processing of rodent samples will be done in a designated closed lab. Only individuals wearing the required protective clothing, and who have

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been certified for respirator use by the Triangle Laboratories Occupational Health-Respiratory Safety Program, are allowed in the room during specimen processing. These room access restrictions are removed after all rodent specimen processing has been completed, working surfaces have been decontaminated, contaminated disposables have been properly stowed, and the room has been aired out for at least 30 minutes.

C. Required protective clothing for specimen processing includes tyvek coveralls, a plastic apron, disposable boot covers, two pairs of latex gloves, non-vented safety goggles, and a properly fit half-mask respirator with organic vapor, acid gas/HEPA filter cartridges.

D. The animals must be packaged as follows:

1. Each individual animal must be packaged separately, even if compositing is required. The chain of custody should contain all compositing information. The individual animals will be ground first in methylene chloride. Compositing will take place once each animal has been ground and can be safely handled.

2. Each individual animal should be wrapped in two layers of aluminum foil, shiny side away from the animal. The outside of the foil encased animal should be labeled with the sample identification number.

3. Each foil encased animal should be placed inside a sealed ziplock bag that is also labeled with the sample identification (ID). This ziplock bag should then be placed inside another ziplock bag such that the seal from the inner bag is at the bottom of the outer ziplock bag. The outer bag should also be labeled with the sample ID.

4. If there are non-rodent samples collected at the same site, these must be shipped ***separately*** from the mice samples. This will allow the laboratory to effectively quarantine the infected samples prior to preparation.

E. For greatest efficiency, two technicians should participate in specimen processing. One technician will be considered "dirty" and will directly handle specimen bags and contents, while the other technician will be considered "clean" and will handle specimen log in forms, blender vessels and tops, and solvent. All direct contact (especially hands) and indirect contact (surfaces of items touched by "dirty" technician) between technicians should be avoided. These roles are not to be switched mid-processing.

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F. After both technicians have donned their protective clothing and the doors to the processing room have been closed, the "dirty" technician should open the shipping container, being careful not to tear his/her latex gloves or tyvek suit.

G. The vertical safety sash on the chemical fume hood should be left in the lowered position to increase the inward linear flow-rate of the fume hood, thereby helping to contain and exhaust any aerosolized virus hazard.

H. The "dirty" technician should sit or stand to the right of the "clean" technician throughout the processing. He/she should remove a specimen from the shipping container, read and/or show the samples identification information to the "clean" technician for log in. The "dirty" technician should not touch the pen or any of the log in supplies.

I. The "clean" technician should load the appropriate amount of methylene chloride solvent into a clean blender vessel and hold it inside the hood work zone within reach of the "dirty" technician. The "dirty" technician should then open and hold the plastic bags containing the specimen and, without directly touching the rodent or the outside of the blender vessel, drop the rodent into the vessel containing the solvent.

J. The "clean" technician should then place a clean lid on the blender vessel, place the vessel on the blender motor, and emulsify the specimen for the appropriate amount of time.

K. The "dirty" technician should then place the empty specimen bags and associated potentially contaminated tags or packing material into the incineration waste container.

L. Steps G-J of this document, above, are then repeated until all specimens have been processed. After emulsification, the "clean" technician is allowed to decant the contents of each blender vessel into another container for storage, and may then remove the vessel from the fume hood work zone if necessary for cleaning and reloading of solvent.

M. If a pair of gloves becomes torn at any time during this procedure, the gloves and any exposed human skin are thoroughly wetted with 10% bleach disinfectant in a continuous hand-washing like manner. If skin has been broken or penetrated by a potentially contaminated object, the gloves are removed after bleach disinfection, and the wound is washed under running water with disinfectant soap for no less than 5 minutes, after which appropriate medical attention is sought. If no injury to the skin has occurred

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a new pair (two pair if necessary) of gloves are donned and the torn pair of gloves are placed in the incineration waste container.

N. All remaining packing material in the shipping container, including any transport ice packs, are then placed into the incineration waste container.

O. When all above steps have been completed, a spray bottle containing 10% bleach disinfectant is used to wet down the sash and horizontal surface of the fume hood, the outside of all other potentially contaminated work surfaces, the outer pair of gloves over the hands of both technicians, the inside and outside of the shipping container, and the spray bottle itself.

P. With gloves wet with bleach disinfectant, the "clean" and "dirty" technicians then grab the outer surface of their left glove with their right double-gloved enabling them to pull off the outer glove on their left hand and drop it into the incineration waste container. The dry inner glove of their left hand is now exposed, which they use to pull off the outer glove on their right hand by slipping a finger under the collared wrist area of the glove as they stand over the incineration waste container.

Q. Wearing the dry inner pair of latex gloves, the technicians should then remove, in the following order, their plastic apron, their disposable boot covers, their tyvek coveralls, their goggles, and their inner gloves (in the manner described above), placing each disposable clothing item into the incineration waste container as it is removed. Any splash contaminated area on their protective clothing should be sprayed with a 10% bleach disinfectant prior to removal. The incineration waste container is then closed and sealed.

R. The floor surface directly around the specimen processing area and shipping container is then sprayed with 10% bleach disinfectant.

S. Wearing their respirators, the technicians then leave the specimen processing room and allow it to ventilate for at least 30 minutes before allowing room re-entry. Outside of the room, they remove their respirators and thoroughly wash their hands with soap and water.

T. The incineration waste container is processed for incineration at the next available opportunity.

U. Subsequent processing of methylene chloride emulsified rodent specimens can then commence without any residual biohazard.

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IV. REFERENCES:

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APPENDIX B

PROCEDURES FOR HANDLING BLOOD AND OTHER POTENTIALLY INFECTIOUS MATERIALS (OPIM)

I. **SCOPE AND APPLICATION:** Procedures delineated in this document are to be applied whenever human blood or OPIM are handled.

II. **SAFETY CONSIDERATIONS:** In the handling of human blood samples or samples containing other human body fluids, it is essential that the precautions delineated below be followed without exception. Precautions must be followed consistently. The approach and procedures delineated below are those recommended by the US Center for Disease Control and Prevention (CDCP). The Hepatitis B (HBV) and the HIV and HTLV-III/LAV viruses (AIDs viruses) appear to be incapable of penetrating the skin. Infection may nevertheless occur as a result of blood or infectious fluids coming in contact with mucus membranes or open wounds. Any individual working with human blood or body fluids must undergo a HBV vaccination program, prove immunity or that a past vaccination is still satisfactory to prevent infection.

III. **PROCEDURE:**

Recommended precautions for handling all blood or other human body fluid samples must be appropriate to prevent contamination, regardless of the knowledge or the lack thereof regarding the infectious state of the media. In the laboratory environment, the greatest risk for transmission involves exposure to a needle or other sharp instrument contaminated with infected blood. Great care must therefore be undertaken to prevent possible transmission via a puncture wound (e.g. needle, sharp glass, etc.)

A. Standard Precautions

1. Handle all human blood and/or body fluids samples as potentially infectious regardless of possible knowledge to the contrary.

2. When working with raw human blood or other body fluids, all work must be done in a hood. Disposable gloves as well as protective eyewear and clothing must be worn. In addition, a disposable mask

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or face shield must be worn whenever procedures, that are likely to generate droplets or aerosols, are performed.

3. Sharp items (needles, glass, etc.) having contacted blood or body fluids should be considered as potentially infective and be handled with extraordinary care. Minimize handling and thus risks associated with such items. Prevent accidental punctures.

4. Disposable syringes, glass pipettes and plastic pipette tips should be placed in puncture resistant containers located as close as possible to the area in which they are being used. If work is being performed in a hood, place the disposal container in the hood, immediately next to or as close as possible to the hood.

5. Hands should be washed immediately and thoroughly with soap and water if contaminated with blood or following glove removal after work with blood or other human body fluid samples. Practice standard good hygiene procedures. Do not handle with contaminated gloves or hand anything a co-worker may handle unprotected (i.e. phone, doorknob, etc.).

6. Spills of blood or other body fluids should be immediately cleaned with soap and water or a household detergent. Individuals performing such cleaning must wear disposable gloves. A freshly prepared solution of sodium hypochlorite (tenfold dilution of off-the-shelf commercial bleach) solution should be used to wipe the area after cleaning in order to thoroughly disinfect the affected area. An alternative to this is to spray the area well with Lysol™.

7. In the event of an exposure (i.e. an accidental puncture wound with a sharp object having contacted blood), immediately wash the area thoroughly with a germicidal soap and water and contact your supervisor for the appropriate follow up procedures.

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TABLE 1: COMMON EMISSIVITY VALUES

Non-Metals		Non-Metals	
Material	**Emissivity**	**Material**	**Emissivity**
Asbestos	.95	Sand	.90
Asphalt	.95	Snow	.90
Basalt	.70	Soil	.90-.98
Brick	.95	Water	.93
Carborundum	.90	Wood, natural	.90-.95
Ceramic	.95	**Metals**	
Clay	.95	**Material**	**Emissivity**
Concrete	.95	Alloy A3003 Oxidized	.30
Cloth	.95	Brass:	
Glass Plate	.85	Burnished	.30
Gravel	.95	Oxidized	.50
Gypsum	.80-.95	Carbon:	
Ice	.98	Unoxidized	.80-.90
Ice Bath	.95	Graphite	.70-.80
Limestone	.98	Iron:	
Organic Materials		Oxidized	.50-.90
(food, plants, oil, some		Rusted	.50-.90
chemicals)	.90-.98	Wrought, dull	.90
Paint (non-Al)	.90-.95	Lead:	
Paper (any color)	.95	Roughened	.40
Plastic (opaque, over			
20 mils)	.95	Steel:	
Rubber	.95	Oxidized	.70-.90
1.			Cold rolled
	.70-.90		

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290

TLI SOP No. DSP105	Version: 16	Effective Date:	03/02/2001
Author:	Don Harvan	Date Written:	January 10, 2001
Authorization: Phil Albro Management		Date Authorized:	02/23/2001

I. **PURPOSE:** This method provides procedures for the extraction of polychlorinated dibenzo-p-dioxin (tetra- through octachlorinated homologues; PCDDs) and polychlorinated dibenzofuran (tetra- through octachlorinated homologues; PCDFs) from solid samples.

II. **SCOPE** It may be applied to the extraction of non-tissue solids, including but not limited to soil, sediment, pulp, sludge, paper and/or cardboard, according to SW-846 Method 8290. Only the extraction procedure is contained in this SOP.

III. **SAFETY CONSIDERATIONS:** Samples may contain harmful substances. Wear labcoat, appropriate eyewear and gloves. Toluene and ethanol are flammable; avoid flames and sparks. The procedure has been validated for either heptane or hexane, since heptane is much less of a health hazard, it should be used instead of hexane whenever possible, but hexane can be used if heptane is unavailable. Do not breathe the vapors, and avoid contact with skin or eyes, as these solvents may cause irritation, nausea, and dizziness. For additional safety information, see the TLI Safety and Health Manual and the appropriate MSDS.

NOTES: **Slight procedural differences exist for specified sample types in this SOP. Be careful not to overlook these differences.**

IV. **REAGENTS:**

A. Heptane- pesticide grade (< 1 ppm residue)

B. Toluene- pesticide grade (< 1 ppm residue)

C. Ethanol- OmniSolve grade

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D. Ethanol/toluene (68/32 v/v) - Take 68 parts (by volume) of Ethanol and mix well with 32 parts (by volume) of Toluene.

V. GLASSWARE PREPARATION

A. All glassware used in this procedure must have been prepared according to glassware washing SOP.

B. The following glassware is needed for each sample:

1. beaker
2. 500 mL
3. forceps
4. spatula
5. thimble holder - acetone rinsed
6. Soxhlet extractor - acetone rinsed
7. thimble - pre-Soxhlet extracted and ready to use

C. If wet sample ≥ 15 g, Soxhlet Dean Stark (SDS) extractor must be used in addition to the Soxhlet extractor.

D. The flask, thimble holder and Soxhlet or SDS extractor must be the same size.

E. If SDS is required, assemble the SDS extractor with stopcock.

F. Rinse all glassware with n-heptane.

G. Label each beaker and flask by placing a color coded sample label on top of a strip of colored lab tape. The colored lab tape is necessary because the adhesive on the color coded labels is difficult to remove from the glass and will cause contamination problems.

H. All flasks require two labels - one on the side of the flask and one on the neck.

VI. EXTRACTION PROCEDURE:

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 16	Date Written: January 10, 2001

A. Plan the extraction batch. An extraction batch can contain up to twenty (20) samples and must include a method blank and at least one(1) Lab Control Spike (LCS). Complete the QC batch form, including all samples and QC samples in the extraction batch.

B. Observe and document the physical appearance and consistency of each sample on form PSTMF 7 for later entry into the wet lab observation log in MILES. Mix the sample well before taking an aliquot for extraction. Make sure homogeneity is maintained--exclude anything that does not constitute sample's natural matrix.

C. Zero balance. Weigh sample and its container.

D. Record the gross weight before on the Sample Preparation Management and Tracking Form (PSTMF 7).

E. Tare the beaker containing the extraction thimble.

F. Weigh into the thimble the amount of sample needed to provide 10g dry weight according to the moisture determination. If the moisture determination has not been completed yet, it is appropriate for the chemist to make an estimate of the amount of sample needed based on its appearance and consistancy.

G. Record the sample weight on the Sample Preparation Management and Tracking Form (PSTMF 7).

(Y)=There is enough sample left to perform re-extraction
(N)=There is NOT enough sample left to perform re-extraction

H. Zero balance. Weigh leftover sample and its container.

I. Record the gross weight "after" on the Sample Preparation Management and Tracking Form (PSTMF 7).

J. Clean the balance by swiping with heptane.

Prepare the blank using pre-extracted G-8 filters for pulps and all <u>dried and ground samples</u>. Prepare the blank using pre-extracted sand for all normal ash/sediment/soil/sludge samples. Weight 10 grams +/- 0.4 g using the same

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matrix as the blank for any laboratory control samples (OPR or LCS and/or LCSD).

L. Prepare the Soxhlet apparatus by:

1. Use a 500 mL setup, place 400 mL toluene* in the flatbottom flask. Add 5-6 Teflon boiling chips in each flask.
2. Place the label containing information for the concentration process on the boiling flask on top of the colored lab tape. Write the solvent used on the flask.
3. Place the thimble holder on top of the flask.

M. ***NOTE:** GP pulp sludge samples require 68:32 ethanol or reagent alcohol/toluene instead of toluene. GP pulp samples require ethanol or reagent alcohol instead of toluene. The volume remains 400 mL for 500 mL setup.

N. Place the thimble with the sample into the thimble holder. Be sure to seat the thimble at the bottom of the holder.

O. **NOTE:** While the Soxhlet apparatus is sitting on the counter, keep the top capped with aluminum foil to avoid contamination.

P. Spike the samples according to the SOP on adding spike standards to samples.

Q. After spiking place a glass wool plug on top of the sample in each thimble. Use pre-extracted glasswool.

R. Place the Soxhlet on top of the thimble holder. If Soxhlet Dean Stark (SDS) is being used, be sure the stopcock is closed.

S. Place the Soxhlet apparatus on the heating mantle and connect the Soxhlet apparatus to the condenser at the ground glass joint. Cap the top of the condenser with aluminum foil.

T. Wrap the thimble holder and Soxhlet extractor with foil. Do not cover the condenser.

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
TLI SOP No. DSP105 Version: 16	Date Written: January 10, 2001

U. **NOTE:** If using ethanol or reagent alcohol as the sole solvent, do not wrap any of the apparatus with aluminum foil.

V. Place a piece of colored tape on the condenser to indicate it is being used for a sample.

W. Turn on the heat to a level at which each unit should be cycling at a rate of five times per hour.

X. Check the units after one-half hour (1/2) hour. Open the aluminum foil on the thimble holder to check the cycling action and close the wrapping. Each unit should be cycling, have no leaks and have sufficient solvent. Make adjustments as necessary.

Y. If adjustments were required, check the units again in one-half hour (1/2) hour. Periodic checks should be made throughout the required time to ensure proper cycling.

Z. If the weighed sample is >30g, drain the water from the SDS halfway through the extraction (i.e., approximately 8 hours after turning on the heaters).

AA. Extract the sample for 16 hours.

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EXTRACTION OF PCDD/PCDF FROM SOLIDS (NOT TISSUE) - 8290	
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EXTRACTION OF DIOXINS/FURANS FROM WATER FOR METHODS 1613, 8290 AND 551		
TLI SOP No. DSP161	Version: 18	Date Revised: October 9, 2001

Author: Patrice L Singletary Effective Date: 3/12/02

Authorization: P. Rogers Date Authorized: 12/31/01
Management

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I. **PURPOSE:** This SOP provides procedures for the extraction of polychlorinated dibenzo-p-dioxin and polychlorinated dibenzofuran (PCDD and PCDF) from water samples.

II. **SCOPE** . It may be applied to the extraction of tetrachlorodibenzo-p-dioxins and tetrachlorodibenzofuran from water in accordance with method 1613, 8290 or 551. See the SOP on Preparation for Extraction of Dioxins / Furans from Water before using the present SOP.

III. **SAFETY CONSIDERATIONS:** Samples may contain harmful substances. Wear labcoat, appropriate eyewear, and gloves. These solvents are flammable; avoid flames and sparks. Do not breathe the vapors, and avoid contact with skin and eyes. They may cause irritation, nausea, dizziness, and, in extreme cases, death. For additional safety and health information, see the TLI Safety and Health Manual and the appropriate MSDS.

IV. **REAGENTS:**

A. HPLC Grade Water

B. Heptane-pesticide grade or better quality, less than 1 ppm residue on evaporation.

C. Sodium sulfate-anhydrous, pesticide grade.

D. Methylene Chloride-pesticide grade or better quality.

E. Toluene-pesticide grade or better quality.

V. **GLASSWARE PREPARATION:**

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A. All glassware used in this procedure must have been prepared according to the SOP entitled "Glassware Cleaning".

B. The following glassware is needed for each sample:

2 L separatory funnel with stopper and Teflon© stopcock or a continuous liquid-liquid extractor

medium glass powder funnel

receiving flask

500 mL extraction flask and regular thimble holder

thimble (pre-Soxlet extracted)

Soxhlet Dean-Stark (SDS) extractor

C. Assemble the separatory funnel(s) with stopcock and stopper.

D. Rinse the following with heptane:

separatory funnel(s) or continous liquid-liquid apparatus

graduated cylinder(s)

all flasks

glass powder funnel(s)

E. While rinsing the separatory funnel(s), close the stopcock and check for leaks. Drain the funnel into a waste container.

F. After rinsing, invert all flasks and allow to fully drain.

G. Plug the powder funnel(s) with pre-cleaned glasswool. Fill the powder funnel 3/4 full with anhydrous sodium sulfate.

VI. PREPARING SAMPLES FOR EXTRACTION:

A. Plan the extraction batch according to the SOP on batching projects. An extraction batch can contain up to twenty (20) samples and must include a method blank, a spike pair, and at least one (1) Lab Control Spike (LCS).Complete the QC batch form, including all samples and QC samples in the extraction batch.

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B. Remove the samples from storage (cooler) and place them on clean lab bench. Allow sufficient time for samples to reach ambient temperature.

C. Check sample IDs on sample containers against IDs on Sample Preparation Tracking and Management Form. If there are any discrepancies contact the Project Scientist for further instructions.

D. Shake each sample bottle for one minute. If the available volume is less than 900 mL, hold the extraction and contact the Project Scientist for further instructions.

Note: An aliquot cannot be taken of an aqueous sample. The entire contents of the bottle must be used.

E. Use 1000 mL of HPLC water for the blank.

F. Record sample volume in the Wet Lab Observation menu in the MILES.

G. Pour the measured sample volume into a 2 L separatory funnel.

H. Keep the labeled graduated cylinders and sample bottles together. These containers will be rinsed with the extraction solvent in a later step.

I. Record pH and observations of physical appearance of samples (i.e. color, viscosity) in the Wet Lab Observation menu in the Miles.

J. Spike samples according to the WAG on adding spike standards to samples.

K. Shake the spiked water samples for 1 minute.

L. If filtration is required, filter each sample using the suction filtering system. If necessary, rinse the sample bottle and separatory funnel with 50 mL HPLC water and also filter. Transfer the filtered sample back into the 2 L separatory funnel.

M. Extract the filters and all filtered solids according to one of the SOP's for extracting solids. Appropriate extraction methods include the accelerated solvent extractor (ASE) or soxhlet extractor.

VII. SEPARATORY FUNNEL EXTRACTION PROCEDURE:

Note: For samples being analyzed by 8290, when the sample is judged to contain 1 percent or more solids, the sample must be filtered through a glass fiber filter that has been rinsed with toluene. If the suspended solids content is too great to filter through the filter, decant, and then filter the aqueous phase. For samples being analyzed by 1613, when the sample contains greater than 1 percent solids, a sample aliquot

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sufficient to provide 10g of dry solids is used. Decant excess water. If necessary to remove water, filter the sample through a glass-fiber filter and discard the aqueous liquid.

A. If it has not already been done in the preparation of the sample for extraction, pour the sample volume into a 2 L separatory funnel. Rinse the sample bottle and filtration flask if used with 120 mL of methylene chloride and transfer to the separatory funnel.

B. Extract the aqueous phase with methylene chloride as follows:

1. Shake the separatory funnel with water sample and methylene chloride for 2 minutes by hand. Be sure to vent the funnel periodically to avoid pressure buildup.

2. Allow the solvent layers to settle for 10 minutes. If emulsions occur, filter the water + emulsion through a G8 filter or glass wool with a methylene chloride rinse. Add the methylene chloride rinses to the methylene chloride extract.

3. Drain the methylene chloride layer through anhydrous sodium sulfate into a receiving flask.

4. Repeat steps above steps two more times using 60 mL methylene chloride for each extraction. A total of three extractions are required for each sample.

5. Rinse the sodium sulfate twice with 10 mL methylene chloride. Collect the methylene chloride rinses in the receiving flask containing the sample extract.

6. Measure the volume used for each sample using a graduated cylinder.

VIII. CONTINUOUS LIQUID-LIQUID EXTRACTION PROCEDURE:

Note: The use of the continuous liquid-liquid extractor is always allowed as an alternative to separatory funnel extraction in method 8290. It is the technique of choice when a given sample type is known to produce difficult emulsions. Use this technique with methods 1613 (B) and 551 whenever a re-extraction is needed because of low recoveries during the separatory funnel extraction step. Be sure to indicate on the extraction form whenever L-L extraction was used.

Note 2: The L-L extractor simply substitutes for a separatory funnel; therefore all considerations of filtering, determination of solids or particulate solids, or water vs.

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sludge described in the SOP on Preparation for Extraction of Dioxins / Furans from Water for Methods 1613, 8290 and 551 still apply.

A. Assemble Glassware

1. Rinse the extractor body and 500 mL flat bottom flask with methylene chloride. Discard rinse as waste.

2. Place 5-6 methylene chloride pre-extracted boiling chips in the flat bottom flask. Add approximately 250 mL of clean methylene chloride to the flask.

3. Add approximately 150 mL of methylene chloride to the extractor body.

4. Attach extractor body to the flask with green plastic clamp.

5. Label flat bottom with solvent, project number and sample number using color labels.

B. Preparation of Water Sample

1. Measure and record 1 liter of Reagent water each for blank and either OPR (1613), or LCS/LCSD (8290) utilizing a 1000 mL graduated cylinder.

2. Measure the volume of sample and transfer the well mixed sample into the extractor body. Record this volume on the sample preparation and tracking form. Note: If the sample does not contain 1000 mL, record the volume it does contain, but make up the difference with Reagent water. The L-L apparatus needs 1000 mL of water to operate correctly. The sample size to use in calculations, however, is the actual sample size not including make-up water.

3. Rinse the sample bottle three times with 20 mL methylene chloride and transfer rinses to the extractor body.

C. Extraction

1. Place Continuous Liquid-Liquid setup on condenser/hot plate and seat securely in place. Use aluminum foil shimmies to align body vertically and to fill any space between hotplate and bottom of the flask.

2. Turn hotplate on to 400 degrees.

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3. Extract for 18 hours.

4. Dismantle setup after they have cooled. Keep only the methylene chloride in the flat bottom flask.

   Discard water and methylene chloride in extractor body as waste.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290 & TO-9	
TLI SOP: DHR182 Version: 9	Date Written: March 20, 2002

Author: Jennifer Hass Effective Date: April 1, 2002

Authorization: R. Rogoski Date Authorized: March 25, 2002
Management

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I. **PURPOSE:** This SOP is intended to describe the procedures to implement Method 8290.

II. **SCOPE:** This method provides procedures for the analysis of polychlorinated dibenzo-p-dioxins (tetra- through octachlorinated homologues; PCDDs), and polychlorinated dibenzofurans (tetra- through octachlorinated homologues; PCDFs) from extracts of samples prepared according to SW 846 Method 8290 and TO-9.

III. **SAFETY CONSIDERATIONS:** The 2,3,7,8-TCDD isomer has been found to be acenegenic, carcinogenic, and teratogenic in laboratory animal studies. Other PCDDs and PCDFs containing chlorine atoms in the 2,3,7,8 positions are known to have toxicities comparable to that of 2,3,7,8-TCDD. Extreme care must be exercised in all handling of extracts and standards in the application of this SOP. For additional safety information, see the TLI Safety and Health Manual and the appropriate MSDSs.

IV. **SAMPLE COLLECTION, PRESERVATION, AND HANDLING:** See TLI SOP No. 5.01 for sample collection, preservation, and handling information.

V. **PROCEDURE:** The High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometer/Data System (GC/MS/DS) equipment utilized for this analysis includes the following:

A. Gas Chromatographs: Hewlett Packard 5890 or 5890 Series II (equipped for temperature programming and capillary columns)

B. High Resolution Mass Spectrometers: VG 70 Series, VG 70-SE Series, VG Autospec.

C. Data Systems: VG Analytical PDP11 with 11-250 software or VAX Alpha with Opus 3.2 software.

D. <u>GC Injection Port</u> - The GC injection port is designed for capillary columns. Typically, 2 µL injection volumes are used unless otherwise noted.

E. <u>Gas Chromatograph/Mass Spectrometer(GC/MS) Interface</u> - The GC/MS interface components can withstand 350°C. The interface has been designed so

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that the separation of 2,3,7,8-TCDD from the other TCDD isomers achieved in the gas chromatographic column is not appreciably degraded. The GC column is fitted directly into the mass spectrometer ion source without being exposed to the ionizing electron beam. Graphite ferrules should be avoided in the injection port because they may adsorb the PCDDs and PCDFs. Vespel™, or equivalent, ferrules are recommended.

F. Mass Spectrometer - The static resolving power of the instrument must be maintained at a minimum of 10,000 (10 percent valley).

G. Data System - A dedicated data system is employed to control the rapid selected-ion monitoring (SIM) process and to acquire the data. Quantitation data (peak areas or peak heights) and SIM traces (displays of intensities of each ion signal being monitored including the lock-mass ion as a function of time) must be acquired during the analyses and stored. Quantitation may be reported based upon computer generated peak areas or upon measured peak heights. The data system is set to acquire data as low as 10 ions in a single scan. Table 1. presents a listing of the ions which are typically monitored. The data system is set to switch to different sets of ions (descriptors) at specified times during an HRGC/HRMS acquisition. The data system provides hard copies of individual ion chromatograms for selected gas chromatographic time intervals. It also acquires mass spectral peak profiles and provides hard copies of peak profiles to demonstrate the required resolving power. Measurements of noise on the base line are performed using the hard copies of individual ion chromatograms provided by the data system.

NOTE: The detector ADC zero setting must be set to allow peak-to-peak measurement of the noise on the base line of every monitored channel and allow for good estimation of the instrument resolving power.

H. GC Columns

1. In order to have an isomer specific determination for 2,3,7,8-TCDD and to allow the detection of OCDD/OCDF within a reasonable time interval in one HRGC/HRMS analysis, the 60 m DB-5 fused silica capillary column is used.

2. The 2,3,7,8-TCDF isomer must be confirmed on a 30 m DB-225 fused silica capillary column, when 2,3,7,8-TCDF is detected on the DB-5 column at a level greater than or equal to the target detection limit.

VI. STANDARDS:

A. Calibration Solutions Six nonane solutions containing 17 unlabeled PCDDs and PCDFs and 18 $^{13}C_{12}$ -labeled PCDDs and PCDFs at known concentrations are used to calibrate the instrument. The analyte concentration ranges are

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homologue dependent, with the lowest values for the tetrachlorinated dioxin and furan (0.5 pg/µL) and the highest values for the octachlorinated congeners (2000 pg/µL) (Table 2.).

B. Recovery Standard Solution - This nonane solution contains two recovery standards, $^{13}C_{12}$-1,2,3,4-TCDD and $^{13}C_{12}$-1,2,3,7,8,9-HxCDD, at a nominal concentration of 100 pg/µL per compound. A 20 µL of this solution is spiked into each sample extract before the HRGC/HRMS analysis.

C. GC Column Performance/Retention Window Check Solution (RTCHK)- This solution contains the first and last eluting isomers for each homologous series from tetra- through heptachlorinated congeners. The solution also contains a series of closely eluting TCDD and TCDF isomers for the purpose of documenting the chromatographic resolution (Table 3).

D. Acceptance Criteria for Newly Prepared Standards - All components and concentrations of each calibration standard, recovery standard, internal standard, and matrix spike solutions are verified prior to use for the analysis of samples. Testing consists of back to back analysis of the "test" solution (the newly prepared solution) and a "control" solution (an independent, second source solution purchased from a qualified vendor). Control solutions are isolated from the production standards in a protected location. Both the control and test solutions are evaluated versus the current continuing calibration standard and versus each other. Each component of the test solution must be within 80 - 120% of the true concentration when calculated versus the control standard. The control standard must be within 80 - 120% of the true value when calculated versus the continuing calibration standard.

Note: The control solution is a second source standard that is purchased ready to use from a qualified vendor such as Cambridge Isotape Laboratories

E. Standards are stored in 1/2 dram amber glass vials at room temperature.

VII. SYSTEM PERFORMANCE CRITERIA

System performance criteria are presented below. It must be documented that all applicable system performance criteria specified in this Section are met before analysis for any sample is performed. Table 4 provides recommended GC conditions that can be used to satisfy the required criteria. During a typical 12-hour analysis sequence, the GC column performance and mass spectrometer resolving power checks must be performed at the beginning of the 12-hour period of operation. A routine calibration verification is required at the beginning and end of each 12-hour period during which samples are analyzed. A method blank or HRGC/HRMS solvent blank is required between a calibration run and the first sample run.

A. GC Column Performance

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1. Inject the column performance check solution described in the standards section of this SOP.Acquire selected-ion monitoring (SIM) data using the equipment and settings described in the equipment section of this SOP.

2. The chromatographic separation between 2,3,7,8-TCDD and the peaks representing any other TCDD isomers or between 2,3,7,8-TCDF and the peaks representing other TCDF isomers must be resolved with a valley of $\leq$ 25% (Fig. 1.), where:

   Valley Percent = (x/y) x 100

   x = height of valley measured between 2,3,7,8-TCDD or 2,3,7,8-TCDF and the closest TCDD or TCDF eluting isomers, and

   y = the peak height of 2,3,7,8-TCDD or 2,3,7,8-TCDF.

3. The acquisition time windows must be set to allow observation of the first and last eluting isomer of each congener. All first and last eluters of a homologous series should be labeled and identified on chromatograms.

B. Mass Spectrometer Performance

1. The mass spectrometer must be operated in the electron ionization mode. It is recommended that the ionization potential be set to optimize sensitivity for the given column flow and source design. A static resolving power of at least 10,000 (10% valley definition) must be demonstrated at appropriate masses before any analyses are performed. Static resolving power checks must be performed at the beginning and at the end of each 12-hour period of operation. It is recommended, however, that a check of the static resolution be made and documented by using the peak matching unit before and after each analysis. Corrective actions must be implemented whenever the resolving power does not meet the requirement.

2. Chromatography time for PCDDs and PCDFs exceeds the long-term mass stability of the mass spectrometer. Because the instrument is operated in the high-resolution mode, mass drifts of a few ppm can have serious adverse effects on instrument performance. Therefore, a mass-drift correction is mandatory. To that effect, use a lock-mass ion from the reference compound Perfluorokerosene (PFK) used for tuning the mass spectrometer and monitor and record the lock-mass ion channel during SIM acquisitions. The level of the reference compound (PFK) metered inside the ion chamber during HRGC/HRMS analyses should be adjusted so that the amplitude of the selected lock-mass ion signal, regardless of

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the description number, does not exceed 10% of the full scale deflection for a given set of detector parameters. Under those conditions, sensitivity changes that might occur during the analysis can be more effectively monitored.

3. Using PFK molecular leak and an appropriate ion within the scan window, tune the instrument to meet the minimum required resolving power of 10,000 (10% valley).

   a) Documentation of the mass spectrometer resolving power is accomplished by recording the peak profile of the high-mass reference signal (m/z 416.9760) obtained during a peak matching experiment by using the low-mass PFK ion at m/z 330.9792 (or lower in mass) as a reference.

   b) The format of the peak profile representative allows manual determination of the peak resolution. The peak width (at 5% peak height) of the high-mass reference ion must not exceed 100 ppm (resolving power: 10,000). Peak width is determined by triangulation with no more than 10% allowance for sampling error. Instrumental ion transmission and resolution will be checked, adjusted and documented in case the resolution is below the minimum required 10,000 resolving power.

VIII. CALIBRATION PROCEDURES:

A. Initial Calibration (ICAL)- Initial calibration of the instrument is required before any samples are analyzed for PCDDs and PCDFs. Initial calibration is also required if any continuing calibration does not meet the required criteria listed in Section VI. B..

1. All six calibration solutions listed in Table 2. must be used for the initial calibration.

2. Tune the instrument with PFK to achieve a static resolving power of at least 10,000 (10% valley) as described in the section on mass spectrometer performance.

3. Inject 2 µL of the GC column performance check solution and acquire SIM mass spectral data –using the equipment and settings described in the equipment section of this SOP. Further analysis must not be performed the column performance criteria listed in the GC column performance section has been met and documented.

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4. Using the same GC and MS conditions that produced acceptable results for the column performance check solution, analyze a 2 µL portion of each of the six calibration solutions with the following requirements.

   a) The ratio of integrated ion current for the ions appearing in Table 5 (homologous series quantitation ions) must be within the indicated control limits (set for each homologous series).

   b) The ratio of integrated ion current for the ions belonging to the carbon-labeled internal, surrogate, alternate, and recovery standards must be within the control limits stipulated in Table 5.

   NOTE: Ion ratios for all 17 native analytes and 18 carbon-labeled internal and recovery standards must be within the specified control limits simultaneously in one run for each of the six (6) calibration standard solutions. If the ion abundance ratios are outside the limits, corrective action must be taken and acceptable abundance ratios achieved before any samples may be analyzed.

   c) For each SICP and for each GC signal representing the elution of a target analyte the signal-to-noise ratio (S/N) must be better than or equal to 10:1.

   d) Referring to Table 6., calculate the 17 relative response factors (RRF) for unlabeled target analytes relative to their appropriate internal standards, according to the following formula:

$$RRF(n) = \frac{A_x \times Q_{is}}{Q_x \times A_{is}}$$

Where:

RRF(n)	=	Analyte RRF
A_x	=	sum of the integrated ion abundances ofthe quantitation ions (Table 1.) for unlabeled PCDDs/PCDFs,
A_{is}	=	sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standards,
Q_{is}	=	quantity of the internal standard injected (pg),
Q_x	=	quantity of the unlabeled PCDD/PCDF analyte injected (pg).

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The RRF(n) is a dimensionless quantity; the units used to express Q_{is} and Q_x must be the same.

e) Calculate the average analyte $\overline{RRF}$ and their respective percent relative standard deviations (%RSD) for the six calibration solutions.

$$\overline{RRF}_{(n)} = \frac{1}{6}\sum_{j=1}^{6} RRF_{(n)j}$$

Where n represents a particular PCDD/PCDF (2,3,7,8 substituted congener), and j is the injection number (or calibration solution number).

f) The relative response factors to be used for the determination of the concentration of total isomers in a homologous series are calculated as follows:

(1) For congeners that belong to a homologous series containing only one isomer (e.g., OCDD and OCDF) or only one 2,3,7,8-substituted isomer (TCDD, PeCDD, HpCDD, and TCDF), the mean RRF used will be the same as the mean RRF calculated for the associated unlabeled target analyte.

NOTE: The calibration solutions do not contain $^{13}C_{12}$-OCDF as an internal standard. This is because a minimum resolving power of 12,000 is required to resolve the $[M+6]^+$ ion of $^{13}C_{12}$-OCDF from the $[M+2]^+$ ion of OCDD (and $[M+4]^+$ from $^{13}C_{12}$-OCDF with $[M]^+$ of OCDD). Therefore, the RRF for OCDF is calculated relative to $^{13}C_{12}$-OCDD.

(2) For congeners that belong to a homologous series containing more than one 2,3,7,8-substituted isomer (PeCDF - two, HxCDF - four, HxCDD - three, HpCDF - two) the mean RRF used for those homologous series will be the average of the mean RRFs calculated for all individual 2,3,7,8-substituted congeners.

NOTE: HRGC/HRMS responses of all isomers in a homologous series that do not have the 2,3,7,8-substitution pattern are assumed to be the same as the responses of one or more of the 2,3,7,8-substituted isomer(s) in that homologous series.

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g) Referring to Table 7. and 8. calculate relative response factors (RRF) and average relative response factors ($\overline{RRF}$) for internal, surrogate, and alternate standards relative to their appropriate recovery standards, according to the following formula:

$$RRF_{(m)} = \frac{A_s \times Q_{rs}}{Q_s \times A_{rs}}$$

$$\overline{RRF}_{(m)} = \frac{1}{6}\sum_{j=1}^{6} RRF_{(m)j}$$

Where:

m	=	congener type,
j	=	injection number,
A_s	=	sum of the integrated ion abundances of the quantitation ions for a given standard,
A_{rs}	=	sum of the integrated ion abundances of the quantitation ions for the appropriate recovery standard,
Q_{rs}, Q_s	=	quantities of, respectively, the recovery standard (rs) and a particular standard injected (pg),
$RRF_{(m)}$	=	relative response factor of a particular standard relative to an appropriate recovery standard, as determined from one injection, and
$\overline{RRF}_{(m)}$	=	calculated mean relative response factor of a particular labeled standard relative to an appropriate recovery standard, as determined from the six initial calibration injections.

5. Acceptance Criteria for Initial Calibration - The criteria listed below for acceptable calibration must be met before any analysis is performed.

a) The percent relative standard deviations for the mean response factors from the 17 unlabeled standards must not exceed ± 20 percent, and those for the labeled reference compounds must not exceed ± 30%.

b) The S/N ratio for the GC signals present in every SICP (including the ones for the labeled standards) must be ≥ 10.

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f) Continuing calibrations analyzed at the end of the 12 hour period which fail by more than 25% RPD for the unlabeled compounds and 35% RPD for the labeled reference compounds must be documented according to the non-conformance SOP.

IX. ANALYSIS

A. A valid analysis of column performance check, calibration and method or instrument blank must have been obtained prior to the analysis of any sample.

B. Remove the sample extract (blown to dryness) or blank from storage. Add 20 µL of recovery standard and mix thoroughly. Care must be taken to coat the walls of the vial several times to dissolve the sample material deposited on the walls during the concentration process.

NOTE: A final volume of 20 µL or more should be used whenever possible. A 10 µL final volume is difficult to handle, and injection of 2 µL out of 10 µL leaves little sample for confirmations and repeat injections, and for archiving.

C. Inject a 2 µL aliquot of the extract into the GC, operated under the GC conditions that have produced acceptable CONCAL and RTCHK results.

D. Acquire SIM data using the same acquisition and mass spectrometer operating conditions previously used to determine the relative response factors.

NOTE: The acquisition period must at least encompass the PCDD/PCDF overall retention time window previously determined. Selected ion current profiles (SICP) for the lock-mass ions (one per mass descriptor) must also be recorded and included in the data package. These SICPs must be true representations of the evolution of the lock-mass ions amplitudes during the HRGC/HRMS run. The analyst may be required to monitor a PFK ion, not as a lock mass, but as a QC ion, in order to meet this requirement. It is recommended to examine the QC ion or lock-mass ion SICP for obvious basic sensitivity and stability changes of the instrument during the GC/MS run that could affect the measurements. Report any discrepancies in the case narrative.

E. Identification Criteria - For a gas chromatographic peak to be identified as a PCDD or PCDF, it must meet all of the following criteria:

1. Retention Times

a) For 2,3,7,8-substituted congeners, which have an isotopically labeled internal or recovery standard present in the sample extract (this represents a total of 10 congeners including OCDD; Table 1.), the retention time (RRT; at maximum peak height) of the sample components (i.e., the two ions used for quantitation

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purposes listed in Table 1.), must be within -1 to +3 seconds of the isotopically labeled standard.

b) For 2,3,7,8-substituted compounds that do not have an isotopically labeled internal standard present in the sample extract (this represents a total of eight congeners; Table 1.), the retention time must fall within 0.005 retention time units of the relative retention times measured in the continuing calibration. Identification of OCDF is based on its retention time relative to $^{13}C_{12}$-OCDD as determined from the 12 hour continuing calibration results.

c) For non-2,3,7,8-substituted compounds (tetra through octa; totaling 119 congeners), the retention time must be within the corresponding homologous retention time windows established by analyzing the RTCHK solution.

d) The ion current responses for both ions used in quantitation (e.g., for TCDDs: m/z 319.8965 and 321.8936) must reach maximum simultaneously (± 2 seconds).

e) The ion current responses for both ions used for the labeled standards (e.g., for $^{13}C_{12}$-TCDD: m/z 331.9368 and m/z 333.9339) must reach maximum simultaneously (± 2 seconds).

2. Ion Abundance Ratios

The integrated ion current for the two ions used for quantitation purposes must have a ratio between the lower and upper limits established for the homologous series to which the peak is assigned (Table 5.).

3. Signal-to-Noise (S/N) Ratio

All ion current intensities must be ≥ 2.5 times noise level for positive identification of a PCDD/PCDF compound or a group of coeluting isomers.

4. Polychlorinated Diphenyl Ether Interferences

In addition to the above criteria, the identification of a GC peak as a PCDF can only be made if no signal having a S/N ≥ 2.5 is detected, at the same retention time (± 2 seconds), in the corresponding polychlorinated diphenyl ether (PCDPE, Table 1.) channel or if the PCDPE signal is less than 10% of the PCDF signal.

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F. Quantitation

1. For gas chromatographic peaks that have met specified criteria calculate the concentration of the PCDD or PCDF compounds using the formula:

$$C_x = \frac{A_x \times Q_{is}}{A_{is} \times W \times \overline{RRF}_{(n)}}$$

Where:

C_x = concentration of unlabeled PCDD/PCDF congeners (or group of coeluting isomers within an homologous series) in pg/g,

A_x = sum of the integrated ion abundances of the quantitation ions (Table 1.) for unlabeled PCDDs/PCDFs,

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standards,

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

W = weight or volume, in grams or liters, of the sample (solid or liquid), and

$\overline{RRF}_{(n)}$ = calculated mean relative response factor for the analyte.

2. Calculate the percent recovery of the nine internal standards measured in the sample extract, using the formula:

$$\text{Internal Standard Recovery} = \frac{A_{is} \times Q_{rs}}{Q_{is} \times A_{rs} \times \overline{RRF}_{(m)}} \times 100$$

Where:

A_{is} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled internal standard,

A_{rs} = sum of the integrated ion abundances of the quantitation ions (Table 1.) for the labeled recovery standard; the selection of the recovery standard depends on the type of congeners (Table 7.),

Q_{is} = quantity, in pg, of the internal standard added to the sample before extraction,

Q_{rs} = quantity, in pg, of the recovery standard added to the cleaned-up sample residue before HRGC/HRMS analysis, and

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$\overline{RRF}_{(m)}$ = calculated mean relative response factor for the labeled internal standard relative to the appropriate (see Table 7) recovery standard.

3. Calculate the percent recovery of seven surrogate and alternate standards using the same equation used to calculate percent recovery of the internal standards.

4. If the concentration in the final extract of any of the fifteen 2,3,7,8-substituted PCDD/PCDF compound exceeds the dynamic range of the instrument (i.e., saturation in a mass channel) then solvent will be added to the extract to bring the signal level into the instrument's dynamic range. Any dilutions must be pre-approved by the client.

5. The total concentration for each homologous series of PCDD and PCDF is calculated by summing up the concentrations of all positively identified isomers of each homologous series. Therefore, the total should also include the 2,3,7,8-substituted congeners. The total number of GC signals included in the homologous total concentration value must be specified in the report.

6. Sample Specific Estimated Detection Limit - The sample specific estimated detection limit (EDL) is the concentration of a given analyte required to produce a signal with a peak height of at least 2.5 times the background signal level. An EDL is calculated for each 2,3,7,8-substituted congener that is not identified, regardless of whether or not other non-2,3,7,8-substituted isomers are present. Two methods of calculation can be used, depending on the type of response produced during the analysis of a particular sample.

   a) Samples giving a response for at least one quantitation ion that is less than 2.5 times the background level.

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The area of the analyte is replaced by the noise level measured in a region of the chromatogram clear of genuine GC signals multiplied by an empirically determined factor. The detection limits represent the maximum possible concentration of a target analyte that could be present without being detected.

$$DL = \frac{2.5 \times H \times Q_{is}}{H_{is} \times RRF_{(n)} \times W}$$

Where:

DL	=	estimated detection limit for a target analyte, expressed in ng or pg.
2.5	=	minimum signal/ noise required for a GC signal to be accepted.
H	=	height of the noise.
H_{is}	=	integrated current of the characteristic ions of the corresponding internal standard.
Q_{is}	=	amount of internal standard added to sample before extraction.
$\overline{RRF}_{(n)}$	=	mean analyte relative response factor from the initial calibration.
W	=	sample weight or volume.

b) Samples characterized by a response above the background level with a S/N of at least 2.5 for both quantitation ions.

When the response of a signal having the same retention time as a 2,3,7,8-substituted congener has a S/N in excess of 2.5 and does not meet ion ratio requirements (Table 5.) calculate the "Estimated Maximum Possible Concentration" (EMPC) using the equation shown at the beginning of this section for the quantitation of PCDD or PCDF compounds except that A_x should represent the sum of the area under the smaller peak and of the other peak area calculated using the theoretical chlorine isotope ratio.

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X. QUALITY CONTROL REQUIREMENTS

A. GC column performance must be demonstrated initially and verified prior to analyzing any sample in a 12-hour period. The GC column performance check solution must be analyzed under the same chromatographic and mass spectrometric conditions used for other samples and standards.

B. Routine calibrations must be performed at the beginning of a 12-hour period after successful mass resolution and GC resolution performance checks and at the end of a 12-hour period following the analysis of samples.

C. The quantitation limits (or target detection limits) will be the following:

1. Quantitation limits for waters (sample size - 1L):

   ≤ 10 ppq TCDD & TCDF

   ≤ 50 ppq Penta - HeptaCDD & CDF

   ≤ 100 ppq OCDD & OCDF

2. Quantitation limits for solids (sample size - 10g):

   ≤ 1 ppt TCDD & TCDF

   ≤ 5 ppt Penta - HeptaCDD & CDF

   ≤ 10 ppt OCDD & OCDF

3. Quantitation limits for air or Wipes (sample size - 1):

   ≤ 50 pg TCDD & TCDF

   ≤ 250 pg Penta - HeptaCDD & CDF

   ≤ 500 pg OCDD & OCDF

D. Method Blank

1. Percent recoveries of all labeled standards should be between 40 - 135%. Method blank with standard recoveries below 40% (but ≥ 25%) is acceptable as long as signal to noise for affected standards is ≥ 10:1.

2. Qunatitation limites (target detection limits) must be met for each analyte.

3. No reportable analytes should be present in the Method Blank. Up to 3 analytes may be present at levels below 1/2 Target Detection Limit (TDL) as long as the compounds were not reported in the previous analysis.

4. For Method Blanks associated with high level samples, the analyte level in the blank must be < 5% of the quantity present in the samples.

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E. LCS/ LCSD and MS/ MSD

1. Percent recoveries of all labeled standards should be between 40 - 135%. LCS/ LCSD or MS/ MSD with labeled standard recoveries below 40% (but ≥ 25%) are acceptable as long as signal to noise for affected standards is ≥ 10:1.
2. Percent recoveries of all analytes should be between 70 - 130%. For up to two analytes, recoveries may be as high as 145% or as low as 60%, as long as the associated relative percent differences (%RPDs) meet criteria.
3. Relative percent difference between LCS and LCSD or MS and MSD should be ≤ 20% for all analytes. For up to two analytes, %RPDs may be higher (up to 35%), as long as the associated percent recoveries meet criteria.
4. If percent recovery or relative percent difference criteria in MS/MSD pair is not met:
   a) Evaluate data for possible matrix influence
   b) Evaluate LCS data for compliance.
   c) If cause of non-compliance is not determined, reextract sample batch.

F. Samples

1. Percent recoveries of all labeled standards should be between 40 - 135%. Samples with standard recoveries below 40% (but ≥ 25%) are acceptable as long as signal to noise for affected standards is ≥ 10:1. In the case of OCDD internal standards, the OCDF analyte must be below TDL also.
2. Specific detection limits must not exceed the target detection limits unless prohibited by a limited sample size or the need for dilution.
3. If the method blank contains reportable analytes but those analytes are not detected in the sample, the sample data may be reported.
4. Samples which fail acceptance criteria listed above or are associated with failing method blank or LCS/ LCSD must be reextracted and reanalyzed. Exceptions may be made for sample matrices which require extreme additional cleanup procedures.

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XI. **REFERENCES:** This method is used for the analysis of sample extracts prepared by any of the following methods, SOP DSP104, DSP105, DSP108, DSP112, DSP115, DSP124, DSP130, DSP131, DSP133, DSP134, DSP150, DSP154, DSP161, DSP218, DSP254, DSP260, DSP261, DSP272, DSP276, DSP278, DSP280, DSP290, and/or DSP294.

XII. **INTERFERENCES**

A. Phthalate interferences are characterized by major quantitative interference in the PeCDF and PeCDD regions. Samples with phthalates receive additional clean up using the alumina clean up detailed in DSP 260D.

B. Interferences caused by the presence of sulfur in a sample are characterized by quantitative interference in the TCDF and TCDD regions. Samples containing sulfur should receive additional clean up using the copper clean up detailed in DSP 277.

C. Interferences caused by the presence of three-ring PAHs are characterized by quantitative interference in the Tetra through HexaCDD and CDF regions. Samples containing three-ring PAHs should receive additional clean up using sulfuric acid, alumina, and/or carbon clean up detailed in DSP 280 and 260D. Samples that contain fluorescing residue and show this pattern of quantitative interference should be cleaned up using the silver nitrate clean up detailed in DSP 260D.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290 & TO-9	
TLI SOP: DHR182 Version: 9	Date Written: March 20, 2002

TABLE 1: ELEMENTAL COMPOSITIONS AND EXACT MASSES OF THE IONS MONITORED BY HRGC/HRMS FOR PCDDs AND PCDFs

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
2	292.9825	LOCK	C_7F_{11}	PFK
	303.9016	M	$C_{12}H_4{}^{35}Cl_4O$	TCDF
	305.8987	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF
	315.9419	M	$^{13}C_{12}H_4{}^{35}Cl_4O$	TCDF (S)
	317.9389	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO$	TCDF (S)
	319.8965	M	$C_{12}H_4{}^{35}Cl_4O_2$	TCDD
	321.8936	M+2	$C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD
	327.8847	M	$C_{12}H_4{}^{37}Cl_4O_2$	TCDD (S)
	330.9792	QC	C_7F_{13}	PFK
	331.9368	M	$13C_{12}H_4{}^{35}Cl_4O_2$	TCDD (S)
	333.9339	M+2	$^{13}C_{12}H_4{}^{35}Cl_3{}^{37}ClO_2$	TCDD (S)
	375.8364	M+2	$C_{12}H_4{}^{35}Cl_5{}^{37}ClO$	HxCDPE
	339.8597	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF
	341.8567	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF
	351.9000	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO$	PeCDF (S)
	353.8970	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O$	PeCDF (S)
	355.8546	M+2	$C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD
	357.8516	M+4	$C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD
	367.8949	M+2	$^{13}C_{12}H_3{}^{35}Cl_4{}^{37}ClO_2$	PeCDD (S)
	369.8919	M+4	$^{13}C_{12}H_3{}^{35}Cl_3{}^{37}Cl_2O_2$	PeCDD (S)
	409.7974	M+2	$C_{12}H_3{}^{35}Cl_6{}^{37}ClO$	HpCDPE
3	373.8208	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF
	375.8178	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O$	HxCDF
	383.8639	M	$^{13}C_{12}H_2{}^{35}Cl_6O$	HxCDF (S)

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290 & TO-9	
TLI SOP: DHR182 Version: 9	Date Written: March 20, 2002

TABLE 1 CONTINUED

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
3 Continued	385.8610	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO$	HxCDF (S)
	389.8157	M+2	$C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD
	391.8127	M+4	$C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD
	392.9760	LOCK	C_9F_{15}	PFK
	401.8559	M+2	$^{13}C_{12}H_2{}^{35}Cl_5{}^{37}ClO_2$	HxCDD (S)
	403.8529	M+4	$13C_{12}H_2{}^{35}Cl_4{}^{37}Cl_2O_2$	HxCDD (S)
	445.7555	M+4	$C_{12}H_2{}^{35}Cl_6{}^{37}Cl_2O$	OCDPE
	430.9729	QC	C_9F_{13}	PFK
4	407.7818	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF
	409.7789	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O$	HpCDF
	417.8253	M	$^{13}C_{12}H^{35}Cl_7O$	HpCDF (S)
	419.8220	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO$	HpCDF (S)
	423.7766	M+2	$C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD
	425.7737	M+4	$C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD
	435.8169	M+2	$^{13}C_{12}H^{35}Cl_6{}^{37}ClO_2$	HpCDD (S)
	437.8140	M+4	$^{13}C_{12}H^{35}Cl_5{}^{37}Cl_2O_2$	HpCDD (S)
	479.7165	M+4	$C_{12}H^{35}Cl_7{}^{37}Cl_2O$	NCDPE
	430.9729	LOCK	C_9F_{17}	PFK
	441.7428	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO$	OCDF
	443.7399	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	OCDF
	457.7377	M+2	$C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD
	459.7348	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD
	469.7779	M+2	$^{13}C_{12}{}^{35}Cl_7{}^{37}ClO_2$	OCDD (S)
	471.7750	M+4	$^{13}C_{12}{}^{35}Cl_6{}^{37}Cl_2O_2$	OCDD (S)

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TABLE 1 CONTINUED

Descriptor Number[b]	Accurate Mass[a]	Ion Type	Elemental Composition	Analyte
4 Continued	513.6775	M+4	$C_{12}{}^{35}Cl_6{}^{37}Cl_2O$	DCDPE
	442.9728	QC	$C_{10}F_{17}$	PFK

a) The following nuclidic masses were used:

H = 1.007825
C = 12.000000
^{13}C = 13.003355
F = 18.9984
O = 15.994915
^{35}Cl = 34.968853
^{37}Cl = 36.965903

S = Labeled Standard
QC = Ion Selected for Monitoring the Instrument Stability During the GC/MS Analysis

b) Descriptor 1 contains mono-, di- and trichlorinated dibenzodioxins and dibenzofurans that are not quantitated by Method 8290.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290 & TO-9	
TLI SOP: DHR182 Version: 9	Date Written: March 20, 2002

TABLE 2: COMPOSITION OF THE INITIAL CALIBRATION SOLUTIONS

Compound	Concentrations (pg/µL) Sol. Number: 1	2	3	4	5	6
Unlabeled Analytes						
2,3,7,8-TCDD	0.5	1	10	50	100	200
2,3,7,8-TCDF	0.5	1	10	50	100	200
1,2,3,7,8-PeCDD	2.5	5	50	250	500	1000
1,2,3,7,8-PeCDF	2.5	5	50	250	500	1000
2,3,4,7,8-PeCDF	2.5	5	50	250	500	1000
1,2,3,4,7,8-HxCDD	2.5	5	50	250	500	1000
1,2,3,6,7,8-HxCDD	2.5	5	50	250	500	1000
1,2,3,7,8,9-HxCDD	2.5	5	50	250	500	1000
1,2,3,4,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,6,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,7,8,9-HxCDF	2.5	5	50	250	500	1000
2,3,4,6,7,8-HxCDF	2.5	5	50	250	500	1000
1,2,3,4,6,7,8-HpCDD	2.5	5	50	250	500	1000
1,2,3,4,6,7,8-HpCDF	2.5	5	50	250	500	1000
1,2,3,4,7,8,9-HpCDF	2.5	5	50	250	500	1000
OCDD	5	10	100	500	1000	2000
OCDF	5	10	100	500	1000	2000
Internal Standards						
$^{13}C_{12}$-2,3,7,8-TCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	100	100	100	100	100	100

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TABLE 2 CONTINUED

Compound	Concentrations (pg/µL) Sol. Number: 1	2	3	4	5	6
Internal Standards Continued						
$^{13}C_{12}$-OCDD	200	200	200	200	200	200
$^{13}C_{12}$-2,3,7,8-TCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8-PeCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	100	100	100	100	100	100
Surrogate Standards						
$^{37}Cl_4$-2,3,7,8-TCDD	0.5	1	10	50	100	200
$^{13}C_{12}$-2,3,4,7,8-PeCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	100	100	100	100	100	100
Alternate Standard						
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	100	100	100	100	100	100
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	100	100	100	100	100	100
Recovery Standards						
$^{13}C_{12}$-1,2,3,4-TCDD	100	100	100	100	100	100
$^{13}C_{12}$-1,2,3,7,8,9-HxCDD	100	100	100	100	100	100

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TABLE 3: GC RETENTION TIME WINDOW DEFINING SOLUTION AND ISOMER SPECIFICITY TEST STANDARD (SECTION IV. C.)

DB-5 COLUMN GC RETENTION TIME WINDOW DEFINING SOLUTION

CDD/ CDF	FIRST ELUTED	LAST ELUTED
TCDF	1,3,6,8	1,2,8,9
TCDD	1,3,6,8	1,2,8,9
PeCDF	1,3,4,6,8	1,2,3,8,9
PeCDD	1,2,4,7,9	1,2,3,8,9
HxCDF	1,2,3,4,6,8	1,2,3,4,8,9
HxCDD	1,2,4,6,7,9	1,2,3,4,6,7
HpCDF	1,2,3,4,6,7,8	1,2,3,4,7,8,9
HpCDD	1,2,3,4,6,7,9	1,2,3,4,6,7,8

DB-5 COLUMN TCDD SPECIFICITY TEST STANDARD

1,2,3,7 + 1,2,3,8 - TCDD
2,3,7,8 - TCDD
1,2,3,9 - TCDD

DB-225 COLUMN TCDF ISOMER SPECIFICTY TEST STANDARD

2,3,4,7 - TCDF
2,3,7,8 - TCDF
1,2,3,9 - TCDF

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TABLE 4: GAS CHROMATOGRAPHY CONDITIONS

Column type	**DB-5**	**DB-225**
Length (m)	60	30
i.d. (mm)	0.25	0.25
Film Thickness (um)	0.25	0.25
Carrier Gas	Helium	Helium
Carrier Gas Flow (mL/min)	1-2	1-2
Injection Mode	⇐ splitless ⇒	
Valve Time (s)	60	60
Initial Temperature (°C)	150	130
Injection Port Temperature (°C)	250	250
Program Temperature	⇐ See Note ⇒	

Note: The GC temperature program is subject to change. Refer to work area guidelines for exact definitions of the current run conditions and descriptor name.

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TABLE 5. ION-ABUNDANCE RATIO ACCEPTABLE RANGES FOR PCDDs AND PCDFs

Number of Chlorine Atoms	Ion Type	Theoretical Ratio	Control Limits	
			Lower	Upper
4	M/M+2	0.77	0.65	0.89
5	M+2/M+4	1.55	1.32	1.78
6	M+2/M+4	1.24	1.05	1.43
6a	M/M+2	0.51	0.43	0.59
7b	M/M+2	0.44	0.37	0.51
7	M+2/M+4	1.04	0.88	1.20
8	M+2/M+4	0.89	0.76	1.02

a) Used only for ^{13}C-HxCDF
b) Used only for ^{13}C-HpCDF

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TABLE 6. UNLABELED ANALYTES QUANTITATION RELATIONSHIPS

Analyte	Standard Used During Quantitation
2,3,7,8-TCDD	$^{13}C_{12}$-2,3,7,8-TCDD
Other TCDDs	$^{13}C_{12}$-2,3,7,8-TCDD
1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,7,8-PeCDD
Other PeCDDs	$^{13}C_{12}$-1,2,3,7,8-PeCDD
1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,7,8,9-HxCDD	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
Other HxCDDs	$^{13}C_{12}$-1,2,3,6,7,8-HxCDD
1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
Other HpCDD	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD
OCDD	$^{13}C_{12}$-OCDD
2,3,7,8-TCDF	$^{13}C_{12}$-2,3,7,8-TCDF
Other TCDFs	$^{13}C_{12}$-2,3,7,8-TCDF
1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,7,8-PeCDF
Other PeCDFs	$^{13}C_{12}$-1,2,3,7,8-PeCDF
1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,7,8,9-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF

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TABLE 6 CONTINUED

Analyte	Standard Used During Quantitation
2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
Other HxCDFs	$^{13}C_{12}$-1,2,3,6,7,8-HxCDF
1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
Other HpCDFs	$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF
OCDF	$^{13}C_{12}$-OCDD

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TABLE 7: INTERNAL STANDARDS QUANTITATION RELATIONSHIPS

Internal Standard	Standard Used During Percent Recovery Determination
$^{13}C_{12}$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-OCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,7,8-TCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

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TABLE 8: SURROGATE/ALTERNATE STANDARDS QUANTITATION RELATIONSHIPS

Surrogate/Alternate/Cleanup Standard	Standard Used During Percent Recovery Determination
$^{37}Cl_4$-2,3,7,8-TCDD	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-2,3,4,7,8-PeCDF	$^{13}C_{12}$-1,2,3,4-TCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-1,2,3,7,8,9-HxCDF	13C_{12}-1,2,3,7,8,9-HxCDD
$^{13}C_{12}$-2,3,4,6,7,8-HxCDF	$^{13}C_{12}$-1,2,3,7,8,9-HxCDD

NOTE : For air samples (PUF and M-0023A) recoveries of surrogate standards are quantitated relative to their appropriate internal standards.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290 & TO-9	
TLI SOP: DHR182 Version: 9	Date Written: March 20, 2002

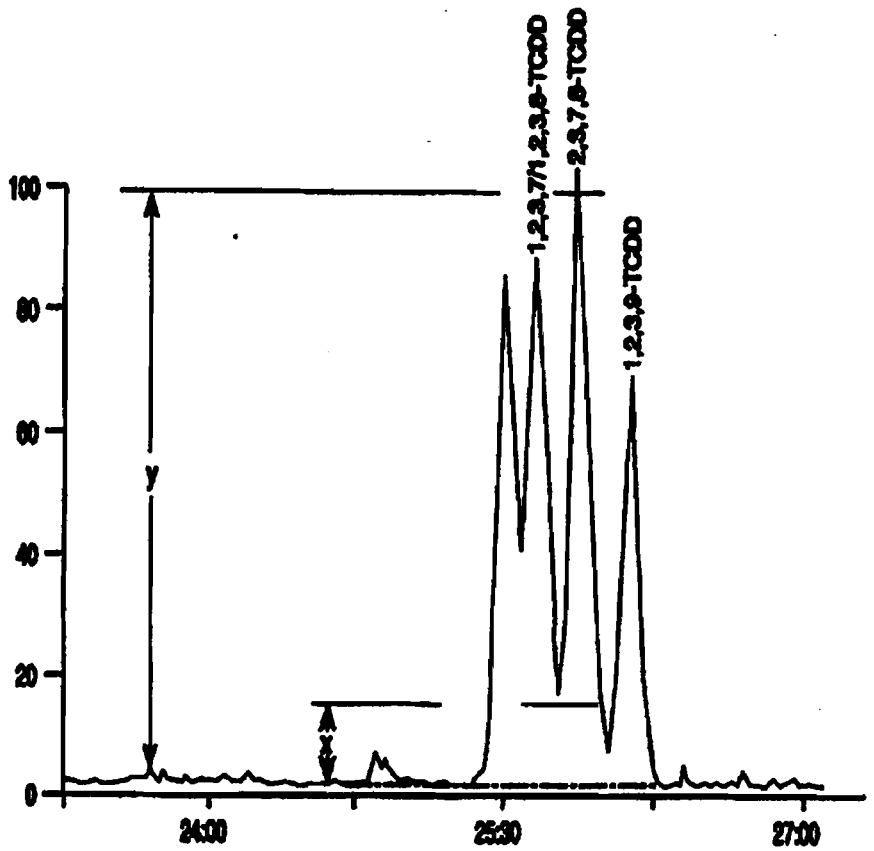

FIGURE 1. Valley between 2,3,7,8 - Tetrachlorodibenzo-p-dioxin and Other Closely Eluting Isomers.

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PCDDs AND PCDFs BY HRGC/HRMS - METHOD 8290 & TO-9	
TLI SOP: DHR182 Version: 9	Date Written: March 20, 2002

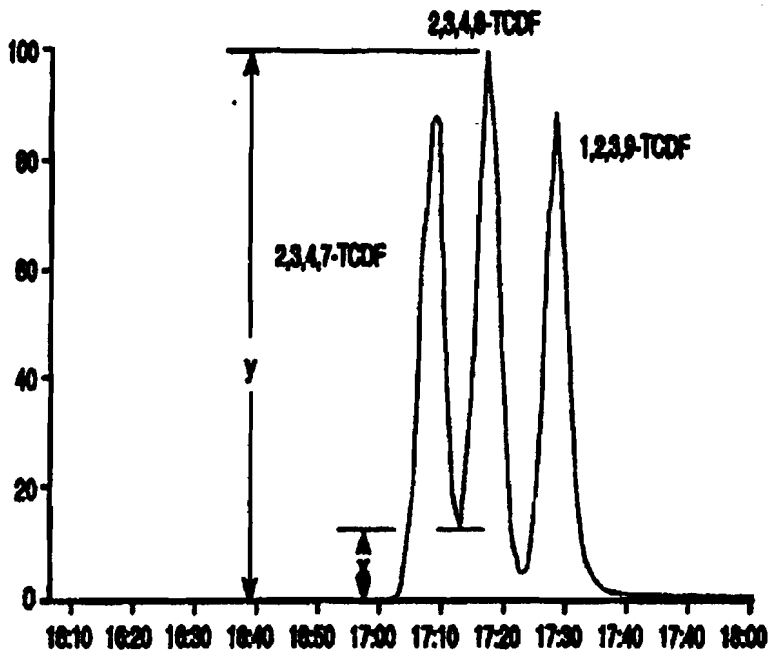

FIGURE 2. Valley between 2,3,7,8 - Tetrachlorodibenzofuran and Other Closely Eluting Isomers.

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APPENDIX 1

Deviations from and improvements to US EPA Method 8290
POLYCHLORINATED DIBENZODIOXINS (PCDDs) AND POLYCHLORINATED DIBENZOFURANS (PCDFs) BY HIGH-RESOLUTION GAS CHROMATOGRAPHY/ HIGH-RESOLUTION MASS SPECTROMETRY (HRGC/ HRMS)
as performed at Triangle Laboratories, Inc.

See Section	
• VI. A. and Table 2.	Triangle Laboratories uses 6 calibration points ranging from 0.5 pg/µL to 200 pg/µL for tetrachlorinated dioxin and furan. Concentrations of penta- through heptachlorinated analytes are 5 times higher than tetra congeners and octachlorinated analytes are 10 times higher (Compare with Table 5., Method 8290).
• VI. A.	Seven more labeled PCDDs/ PCDFs are used as surrogate and alternate standards to provide additional quality control information.
• VI. A.	The 1,2,3,6,7,8 - hexachlorodibenzofuran (HxCDF) is used as an internal standard instead of 1,2,3,4,7,8 - HxCDF. Triangle Laboratories uses 1,2,3,4,7,8 - HxCDF as a surrogate standard.
• VI. A.	Each carbon-labeled standard in the initial calibration solution has a concentration of 100 pg/µL except $^{13}C_{12}$ - OCDD, which is at 200 pg/µL. Concentrations of $^{13}C_{12}$ - labeled standards in sample fortification solutions are the same as in the initial calibration solution (See section 5.9., Method 8290).
• VII.	The method blank does not need to be analyzed on each analysis clock that samples are analyzed. Once a valid analysis is provided for the method blank it may be replaced with HRGC/ HRMS solvent blank (See section 8.2., Method 8290).
• VII. B. 3. a)	Documentation of the mass spectrometer resolving power is accomplished by recording the peak profile of m/z 416.9760 and 330.9792 (See section 8.2.2.3., Method 8290).
• IX. F. 6. a)	To calculate the sample specific Estimated Detection Limit, noise equivalent area is used instead of noise height (See section 7.9.5.1.1., Method 8290).
• XI. D. - F. 1.	The Internal Standard recoveries in all QC samples and field samples are considered valid as long as the signal to noise ratio is greater than 10:1, the recovery is ≥ 25% and target detection limits for the analytes are met (See section 8.4., Method 8290).
• X. E. 2.	For up to two analytes in LCS/ LCSD and MS/ MSD, recoveries may be as high as 145% or as low as 60%, as long as the associated relative percent differences (%RPDs) meet criteria.

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APPENDIX 1 Continued

- X. E. 3. For up to two analytes in LCS/ LCSD and MS/ MSD, %RPDs may be higher (up to 35%), as long as the associated percent recoveries meet criteria.

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DATA PACKAGE ASSEMBLY AND SHIPPING			
TLI SOP No. DPA104	Version: 12	Effective Date:	11/30/98
Author:	David Chu	Date Written:	October 1, 1998
Authorization: Phil Albro Management		Date Authorized:	11/20/98

I. **SCOPE AND APPLICATION:** The purpose of this SOP is to describe the procedures to follow when assembling and shipping all data packages at Triangle Laboratories, Inc. The deliverables of the standard data package contain an identification document stating that Triangle Laboratories, Inc. produced the results.

II. **PROCEDURE:**

A. Assemble the data package, including: the address documentation, the signed case narrative, Triangle Labs list of certifications, document control or project description section, all Sample Data Summary sheets with chromatograms and calibration data for each project. Paginate everything that is going to the client. Go to the last page of the Case Narrative and, in the space provided, enter the total number of pages in the document. Unless the client has requested otherwise, send them the original case narrative and the copy of other deliverables.

NOTE: To obtain the address documentation, go into MILES and Select "F", enter project number, printout destination, Select "M" for Marked for shipment. If it is to be shipped out the door, then type "Y" for transfer to archive. If not, type "N".

B. For high resolution data packages only, locate all calibration data pertaining to the project. Refer to the Reporting Option located in the Reporting Requirements of the Sample Tracking and Project Management Form. Copy only summary sheets for CONCALs and ICALs to send to customer. Some clients request full calibration data which includes summary data sheets and chromatograms for CONCALs, ICALs and retchecks. Calibration data for all other projects should be furnished.

C. Before copying data package, go to the MILES system. At the main menu, select "F" for Shipping/Archiving. On Shipping/Archiving Information Sub-menu, select "Sample shipment Update/Inquiry."

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DATA PACKAGE ASSEMBLY AND SHIPPING					
TLI SOP No.	DPA104	Version:	12	Date Written:	October 1, 1998

D. Enter project number, extension (if applicable) and "M" for marked for shipment, "T" for transfer to archive. Also enter password for this screen.

E. The samples for this project will appear on the screen. The project must be in shipping (D-SH, O-SH, I-SH, S-SH) in order to perform this function. If not, go back to the shipping sub-menu and select option "Production By Project and/or Worklist." Transfer project to shipping.

F. All samples for project will be tagged if project is in shipping. Press <F10> to transfer shipping information to accounting. The computer will issue a printout with all the shipping information including price, address, and special instructions.

G. Follow all instructions closely. Copy data packages according to instructions listed on the printout.

H. The computer will automatically archive all projects if you indicate it to do so by typing "Y".

I. See SOP on shipping projects.

NOTE: When copying BNA projects, set the copier on 93% reduction to avoid chopping off the interim.

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EXTRACTION OF PCDD/PCDF FROM WATER FOR METHODS 1613, 8290 AND 551

TLI SOP No.	DSP161 Version: 16	Effective Date:	October 23, 1998
Author:	Phil Albro	Date Written:	October 2, 1998
Authorization:	Phil Albro Management	Date Authorized:	10/18/98

I. **SCOPE AND APPLICATION:** This SOP provides procedures for the extraction of polychlorinated dibenzo-p-dioxin and polychlorinated dibenzofuran (PCDD and PCDF) from water samples in accordance with methods 1613 or 8290. It may also be applied to the extraction of tetrachlorodibenzo-p-dioxins and tetrachlorodibenzofuran from water in accordance with method 551. See the SOP on Preparation for Extraction of PCDD/PCDF from Water before using the present SOP.

II. **SAFETY CONSIDERATIONS:** Samples may contain harmful substances. Wear labcoat, appropriate eyewear, and gloves. These solvents are flammable; avoid flames and sparks. Do not breathe the vapors, and avoid contact with skin and eyes. They may cause irritation, nausea, dizziness, and, in extreme cases, death. For additional safety and health information, see the TLI Safety and Health Manual and the appropriate MSDS.

III. **REAGENTS:**

A. Organic-free reagent water- Either Dracor or Fisher HPLC Grade

B. Heptane-pesticide grade or better quality, less than 1 ppm residue on evaporation.

C. Sodium sulfate-anhydrous, pesticide grade.

D. Methylene Chloride-pesticide grade or better quality.

E. Toluene-pesticide grade or better quality.

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IV. GLASSWARE PREPARATION:

A. All glassware used in this procedure must have been prepared according to the SOP entitled "Glassware Cleaning".

B. The following glassware is needed for each sample:

2 L separatory funnel with stopper and Teflon© stopcock

A. 1000 mL graduated cylinder

medium glass powder funnel

250 mL round bottom flask

500 mL extraction flask and regular thimble holder (pre-Soxlet extracted).

Soxlet extractor (pre-Soxlet extracted).

Soxhlet Dean-Stark (SDS) extractor - pre-soxhlet extracted and ready for use

A. Assemble the separatory funnel(s) with stopcock and stopper.

B. Rinse the following with heptane:

separatory funnel(s)

graduated cylinder(s)

all flasks

glass powder funnel(s)

A. While rinsing the separatory funnel(s), close the stopcock and check for leaks. Drain the funnel into a waste container.

B. After rinsing, invert all flasks and allow to fully drain.

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C. Plug the powder funnel(s) with pre-cleaned glasswool. Fill the powder funnel 3/4 full with anhydrous sodium sulfate.

I. **SEPARATORY FUNNEL EXTRACTION PROCEDURE:**

A. If has not already been done in the preparation of the sample for extraction, pour the sample volume into a 2 L separatory funnel. Rinse the sample bottle with 60 mL of methylene chloride and transfer to the separatory funnel.

B. Extract the aqueous phase with methylene chloride as follows:

1. Shake the separatory funnel with water sample and methylene chloride for 5 minutes using Glas-Col Automatic Shaker (or 2 minutes by hand). Be sure to vent the funnel periodically to avoid pressure buildup.

   Note: If using the Gas-Col shaker, set the timer for 5 minutes and turn the power on. Slowly increase the speed to 40 or the maximum speed possible without displacing any sample through the vent.

   Allow the solvent layers to settle for 10 minutes. If emulsions occur, filter the water + emulsion through a G8 filter or glass wool with a methylene chloride rinse. Add the methylene chloride rinses to the methylene chloride extract.

2. Drain the methylene chloride layer through anhydrous sodium sulfate into a 250 mL flask.

3. Repeat steps 1-3 two more times using 60 mL methylene chloride for each extraction. A total of three extractions are required for each sample.

1. Rinse the sodium sulfate twice with 10 mL methylene chloride. Collect the methylene chloride rinses in the 250 mL flask containing the sample extract.

I. **EXTRACTING THE FILTER:**

A. Soxhlet extract the filters, glasswool used to break emulsions, and all filtered solids as follows:

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1. Place filters in a toluene pre-Soxhlet extracted thimble (500 mL size).

2. For methods 1613 and 8290, add 375-400 mL of toluene to the 500 mL boiling flask; For method 551, use 400 mL of ethanol: toluene 68:32 (v/v) to the 500 mL flask.

3. Add 5-6 Teflon boiling chips to the boiling flask.

4. Place the label containing information for the concentration process on the boiling flask on top of colored lab tape. Write solvent used on flask with marker.

   a) Do not place set up on hotplates until extraction of the water phase is completed to insure no additional emulsions need to be filtered and the filter or glasswool placed into the thimble with the first filter.

2. Extract using pre-Soxhlet extracted Soxhlet apparatus for 16 hours.

3. After extraction, let cool and ensure the label indicating that this extract is to be combined with the methylene chloride extract from the water phase is still intact.

II. CONTINUOUS LIQUID-LIQUID EXTRACTION PROCEDURE:

Note: The use of the continuous liquid-liquid extractor is always allowed as an alternative to separatory funnel extraction in method 8290. It is the technique of choice when a given sample type is known to produce difficult emulsions. Use this technique with methods 1613 (A or B) and 551 whenever a re-extraction is needed because of low recoveries during the separatory funnel extraction step. Be sure to indicate on the extraction form whenever L-L extraction was used.

Note 2: The L-L extractor simply substitutes for a separatory funnel; therefore all considerations of filtering, determination of solids or particulate solids, or water vs. sludge described in the SOP on Preparation for Extraction of PCDD/PCDF from Water for Methods 1613, 8290 and 551 still apply.

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EXTRACTION OF PCDD/PCDF FROM WATER FOR METHODS 1613A 8290 AND 551					
TLI SOP No.	DSP161	Version:	16	Date Written:	October 2, 1998

A. Assemble Glassware

1. Using pre-extracted glassware, rinse the extractor body and 500 mL flat bottom flask with methylene chloride. Discard rinse as waste.

2. Place 5-6 methylene chloride pre-extracted boiling chips in the flat bottom flask. Add approximately 250 mL of clean methylene chloride to the flask.

3. Add approximately 150 mL of methylene chloride to the extractor body.

4. Attach extractor body to the flask with green plastic clamp.

5. Label flat bottom with solvent, project number and sample number using color labels.

B. Preparation of Water Sample

1. Measure and record 1 liter of Reagent water each for blank and either OPR (1613), or LCS/LCSD (8290) utilizing a 1000 mL graduated cylinder.

2. Measure 1000 mL of well mixed sample and transfer to the extractor body. Record this volume on the sample preparation and tracking form. Note: If the sample does not contain 1000 mL, record the volume it does contain, but make up the difference with Reagent water. The L-L apparatus needs 1000 mL of water to operate correctly. The sample size to use in calculations, however, is the actual sample size not including make-up water.

3. Rinse the 1000 mL graduated cylinder three times with 20 mL methylene chloride and transfer rinses to the extractor body.

C. Extraction

1. Place Continuous Liquid-Liquid setup on condenser/hot plate and seat securely in place. Use aluminum foil shimmies to align body vertically and to fill any space between hotplate and bottom of the flask.

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EXTRACTION OF PCDD/PCDF FROM WATER FOR METHODS 1613A 8290 AND 551	
TLI SOP No. DSP161 Version: 16	Date Written: October 2, 1998

2. Turn hotplate on to 400 degrees.

3. Extract for 18 hours.

4. Dismantle setup after they have cooled. Keep only the methylene chloride in the flat bottom flask.

5. Discard water and methylene chloride in extractor body as waste.

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Quality Assurance and Control Plan

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

March 28, 2001

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J Maegerlein
Name	Michelle L. Spellman
Title	Quality Assurance Officer
Signature	Michelle L. Spellman

MASTER

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CODE 405 3

I. SCOPE AND APPLICATION

The Explosive Sciences Branch (ESB), Code 4052 of the Crane Division, Naval Surface Warfare Center provides scientific laboratory support to the various military services and private constituents. Analysis is normally performed in a laboratory environment. Explosive work is performed both in the laboratory and in remote locations when necessary.

The overall Quality Assurance objective of the laboratory is to develop and implement procedures that will provide sound measurements. This Quality Assurance/Quality Control (QA/QC) Plan is intended to provide the necessary guidance for the laboratory in the development of data that is technically sound and legally defensible. Through teamwork and accountability, the ESB laboratory strives to meet and exceed customer expectations per the Code 405 Quality Plan.

II. SUMMARY OF METHOD

A. Regulations

This method is consistent with ISO 9000 standard requirements and all state and federal testing, safety, and environmental requirements. The table below outlines the branch's mechanism for addressing each element of the standard:

Table 1: Code 4052 ISO Standard Assessment

Element	Applicable Procedure
4.1 Management Responsibility	CR4052-PD-0001
4.2 Quality System	CR4052-PD-0001
4.3 Contract Review	CR4052-PD-0350
4.4 Design Control	N/A
4.5 Document and Data Control	CR4052-PD-0100
4.6 Purchasing	CR4052-PD-0220
4.7 Control of Customer-Supplied Product	CR4052-PD-0500, Method PD's
4.8 Product ID and Traceability	CR4052-PD-0500, CR4052-PD-0150, CR4052-PD-0600
4.9 Process Control	CR4052-PD-0200, Method PD's
4.10 Inspection and Testing	CR4052-PD-0600, CR4052-PD-0500, CR4052-PD-0450
4.11 Control of inspection, measuring, test equip	CR4052-PD-0250
4.12 Inspection and Test Status	N/A
4.13 Control of Nonconforming Product	CR4052-PD-0600
4.14 Corrective and Preventative Action	Code 405 Procedure
4.15 Handling, storage, packaging, preservation and delivery	N/A
4.16 Control of quality records	CR4052-PD-0150
4.17 Internal Quality Audits	Navy Instruction 4855.12A
4.18 Training	CR4052-PD-0300
4.19 Servicing	N/A
4.20 Statistical Techniques	CR4052-PD-0230

B. Objectives

The objective of the laboratory quality assurance program is to assure the accuracy and precision, as well as the reliability of laboratory results produced for our customers, or at the request of regulatory or accrediting bodies. Management, administrative, statistical, investigative, preventive, and corrective techniques will be employed to maximize reliability of the data.

Specific objectives are:

1. To establish the level of quality of the laboratory's routine performance as a baseline against which to measure the effectiveness of quality improvement efforts.

2. To monitor the routine operational performance of the laboratory through participation in appropriate Performance Evaluation programs and to provide for corrective actions as necessary.

3. To improve and validate laboratory methodologies by performing method validation studies.

C. Organization and Responsibility

This section is designed to identify the responsibilities of Quality Assurance within the overall structure of the laboratory organization. Other position descriptions are found in personnel department files and in the training procedures.

The direct and ultimate responsibility for assuring data quality at the ESB laboratory rests with the ESB Manager. The chemist/laboratory analyst will provide the first level of data review in the laboratory, however, the ESB Manager provides the final quality assurance/control of all data gathered or reported for compliance testing. Results may also be reviewed by the customer, regulating agency, or certification body upon request.

All personnel shall strive to meet the requirements of the QA programs. All personnel shall have access to procedure and technical documents and are encouraged to discuss the contents with the management at any time.

CR4052-PD-0001(1) provides an organizational chart within the Explosive Sciences Branch.

1. Explosive Sciences Branch Manager

The Explosives Sciences Branch Manager is primarily responsible for ensuring the QA/QC program within the laboratory is in compliance and data quality adheres to this compliance. In delegating QA responsibilities/authorities, the ESB Manager depends on the Quality Assurance Officer to coordinate all necessary QA requirements. The ESB Manager is responsible for assuring that quality planning is developed, implemented, monitored and updated. The ESB Manager maintains training records and documents for each analyst in all methods in which the analyst is proficient and capable of performing.

2. Quality Assurance Officer

The Quality Assurance Officer is responsible for the performance of the ESB Quality Control program and for taking or recommending measures to ensure the fulfillment of the quality objectives of management while ensuring continued accuracy and precision of the data produced. Duties include but are not limited to the following:

a. Monitors quality control programs, including statistical procedures and techniques, which will enable the laboratory to meet desired quality standards; and advises and assists management in the installation, staffing, and supervision of such programs.

b. Monitors quality control activities of the laboratory to determine conformance with authorized policies and procedures and with sound practice; and makes appropriate recommendations for correction and improvement as may be necessary.

c. The QAO should prepare an annual written Quality Assurance Summary Report.

d. Seeks out and evaluates new technology, methods, and equipment in the field of quality control and recommends means for their application wherever advisable.

e. Prepares for internal and external audits.

f. Advises the purchasing agents with regard to the quality of purchased equipment, materials, reagents, and chemicals.

g. Provides information to the ESB Manager for Management Reviews.

h. Initiates an in-house proficiency evaluation program involving blind samples as required.

i. Maintains appropriate paper copies of procedures and attachments.

2. ESP Laboratory Manager

Duties of the Laboratory Manager include but are not limited to the following:

a. Direct and oversees all laboratory personnel in the enactment of the required analytical procedures and necessary quality control activities.

b. Assists the Quality Assurance Officer in fulfilling the objectives of the Quality Assurance Plan.

c. Reviews the Annual Quality Assurance Summary Report and addresses any outstanding deficiencies.

d. Assures that a complete set of controlled procedures is maintained for all test procedures used by the ESB laboratory.

e. Ensures that only analysts who have completed training conduct analytical methods independently. An analyst in training is directly supervised by an analyst who has completed training.

4. Chemist/Laboratory Analyst

Duties of the Chemist/Laboratory Analyst include but are not limited to the following:

a. Maintains proper log-in and sample tracking documents and active chain-of-custody files.

b. Provides for appropriate disposal of all samples after completion of the analysis.

c. Performs quality control on all samples analyzed utilizing appropriate procedures and providing the necessary documentation as required by the department procedures.

d. Carries out quality control programs, including statistical procedures and techniques, which will enable the laboratory to meet desired quality standards and advise and assist management in the installation, staffing, and supervision of such programs.

e. Monitor quality control activities of the laboratory to determine conformance with authorized policies and procedures and with sound practice; and make appropriate recommendations for correction and improvement as may be necessary.

5. Document Control Coordinator

Duties of the Document Control Coordinator include but are not limited to the following:

a. Maintains controlled electronic procedure copies.

b. Alerts Quality Assurance Officer of required procedure reviews.

III. QUALITY ASSURANCE

The ESB is committed to continuous quality improvement through identifying and managing variation within each analytical process. The policy of the ESB laboratory is to apply the quality system requirements to all testing and analytical activities and includes the following:

A. Quality activities shall emphasize the prevention of quality problems rather than detection and correction of problems after they occur. Periodic internal audits will be conducted and control charts maintained. The laboratory shall proactively identify methods or technology to improve the quality of services provided.

B. All employees engaged in making decisions affecting the quality of laboratory output shall undergo training programs designed to be commensurate with their positions, duties, and responsibilities.

C. The laboratory shall use a single approved analytical test method for each parameter tested, consistent with customer needs for precision, accuracy, and sensitivity, in compliance with State and Federal regulatory programs.

D. The laboratory shall retain copies of all tracking, test and analytical reports in a manner and for a period specified by regulatory or accrediting bodies.

E. Each analyte and associated analytical method requested for certification/accreditation will have an initial demonstration of capability (IDC) performed before any actual sample analysis takes place.

F. The laboratory shall have a comprehensive calibration program involving all instrumentation used for making determinations, the results of which are reported.

G. The laboratory shall practice a supply management program to ensure sufficient supply availability and quality.

H. The laboratory shall participate in an inter laboratory testing program as prescribed by the cognizant certification authority, accrediting organization, or upon customer request.

I. The laboratory shall practice a preventative maintenance program to ensure maximum equipment operating efficiency.

J. The laboratory shall report results in a format and timeframe consistent with consumer requirements.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Quality Assurance Summary Report

On an annual basis, the Quality Assurance Officer will prepare a Quality Assurance Summary Report for review by the ESB Manager. The ESB will use this information to establish priorities and modify this procedure, as required. The report will include the following information:

1. Date of last review of this Quality Assurance Summary Report.

2. A statistical summary of any certification testing including a comparison of blanked or spiked samples.

3. A list of qualified laboratory personnel and the test methods each is certified to perform, including date of most recent certification (see training procedures).

4. Status of annual quality training.

5. Preventative maintenance status.

6. Continuous improvement plan status.

7. A summary of the resolution of any and all corrective actions, preventative actions, audits, and SOP reviews for the previous year.

B. Control Plan

The Code 4052 Control Plans are attached. Separate control plans are included for the different types of analysis performed by the ESB.

C. Quarterly Branch Reviews

The Code 4052 Branch will review quality issues on a quarterly basis. Content of the review will be similar to that of the annual Quality Assurance Summary Report. Notes from these reviews will be taken and maintained per Record Retention, CR4052-PD-0150(1).

VIII. DATA ANALYSIS AND REPORTING

None.

IX. ATTACHMENTS

CR4052-PD-001(1) Code 4052 Organizational Chart, 11/1/00

CR4052-PD-0001(2) Code 4052 Control Plan: NPDES Water, 3/28/01

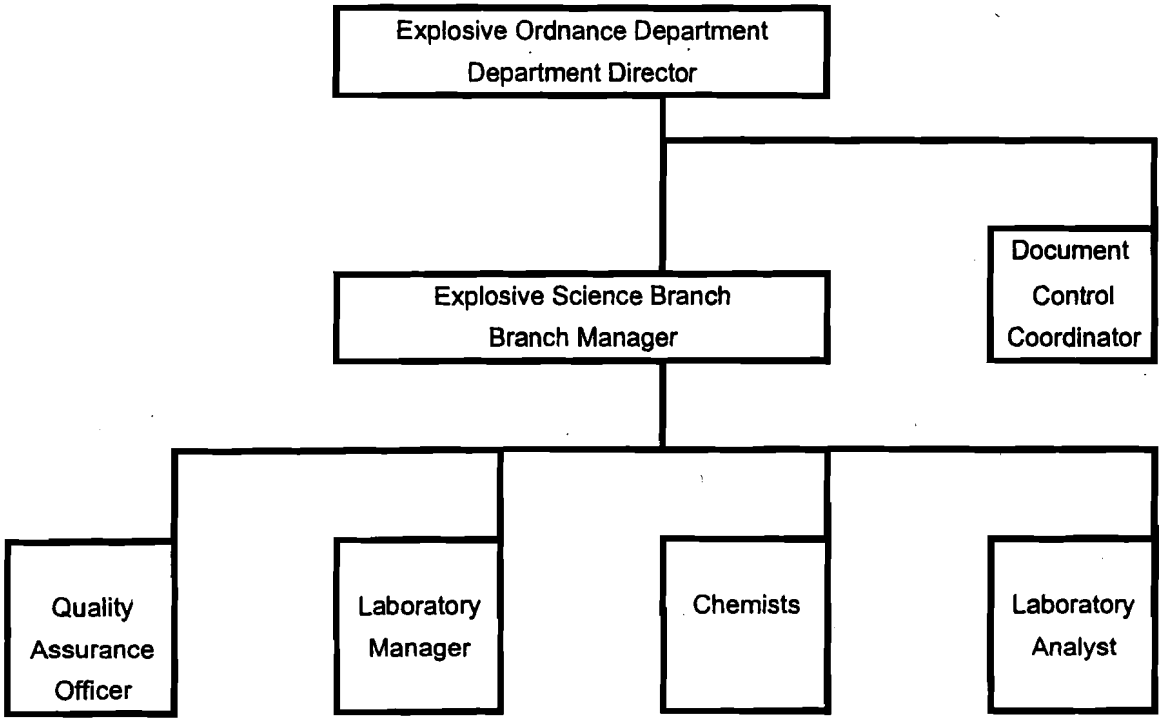
X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	11/1/00	Original issue.	MLS
1	3/28/01	Added Table 1. Updated attachment (2). Added Branch Quarterly Review process.	MLS

Code 4052 Organizational Chart



Code 4052 Control Plan: NPDES Water

Process Step	Operation Description		Characteristic: Sample	Characteristic: Test	Tolerance	Measure	Reaction Plan
.. Prepare Laboratory	1.1	Ensure sufficient supply stock to perform routine testing.	Raw materials known quality.	None.	Chemical Supply Management, CR4052-PD-0220	N/A	Refer to Chemical Supply Management CR4 PD-0220 if material is accepted on an "excep basis.
	1.2	Ensure equipment calibrated.	None.	Results suspect.	Calibration, CR4052-PD-0250	Calibrate equipment prior to each test according to test procedure.	Refer to Calibration CR4052-PD-0250 an Preventative Maintenance CR4052-PD-02
	1.3	Ensure analyst properly trained.	Sample preparation compromised.	Test run improperly.	Training, CR4052-PD-0300; Training: Water, CR4052-PD-0310	Performance evaluations.	Refer to Performance Evaluations, CR4052-PD-0450
2. Receive Sample	2.1	Document and Classify Customer Name, Tests Required, Sample Type	Sample mis-labeled.	Improper test run.	Chain of Custody, CR4052-PD-0500	N/A	Improper test, repeat test procedure.
3. Prepare Sample	3.1	Cadmium, Copper, Lead, Silver, Chromium, Nickel, Zinc, Molybdenum, Potassium	Improper preparation.	N/A	Sample Preparation, CR4052-PD-1000	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	TKN	Improper preparation.	N/A	TKN, CR4502-PD-1210	N/A	If improper standard prepared, repeat preparation procedure.
Select appropriate test:	3.1	T-Phosphorous	Improper preparation.	N/A	Total Phosphorous, CR4052-PD-1220	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Ammonia-N	Improper preparation.	N/A	Ammonia-N, CR4052-PD-1230	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Total Solids	Improper preparation.	N/A	Total Solids, CR4052-PD-1240	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Arsenic, Selenium	Improper preparation.	N/A	Arsenic/Selenium, CR4052-PD-1200	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Mercury	Improper preparation.	N/A	Mercury, CR4052-PD-1300	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Hexavalent Chromium	Improper preparation.	N/A	Hexavalent Chromium, CR4052-PD-1400	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	E-Coli - Wastewater	Improper preparation.	N/A	E-coli in Wastewater, CR4052-PD-1500 Sterilization Procedure, CR4052-PD-0710	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	HMX, RDX, TNT	Improper preparation.	N/A	HMX, RDX, TNT, CR4052-PD-1600	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Nitrate-N	Improper preparation.	N/A	Nitrate - N, CR4052-PD-1700	N/A	If improper standard prepared, repeat preparation procedure.
	3.1	Oil & Grease	Improper preparation.	N/A	Oil and Grease, CR4052-PD-1800	N/A	If improper standard prepared, repea preparation procedure.
	3.1	T. Cyanide	Improper preparation.	N/A	Total Cyanide, CR4052-PD-1900	N/A	If improper standard prepared, repea preparation procedure.
4. Test Sample	4.1	Cadmium, Copper, Lead, Silver, Molybdenum	Damaged sample.	Invalid test.	Graphite Furnace, CR4052-PD-1100 Flame AA, CR4052-PD-1150	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run test.
	4.1	Chromium, Nickel, Zinc, Potassium	Damaged sample.	Invalid test.	Flame AA, CR4052-PD-1150	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run test. Refer to Performance Evaluation, CR4052-PD-0450
Select appropriate test:	4.1	Arsenic, Selenium	Damaged sample.	Invalid test.	Arsenic/Selenium, CR4052-PD-1200	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test.
	4.1	TKN	Damaged sample.	Invalid test.	TKN, CR4502-PD-1210	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test.
	4.1	T-Phosphorous	Damaged sample.	Invalid test.	Total Phosphorous, CR4052-PD-1220	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test.
	4.1	Ammonia-N	Damaged sample.	Invalid test.	Ammonia-N, CR4052-PD-1230	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run test. Refer to Performance Evaluation, CR4052-PD-0450
	4.1	Total Solids	Damaged sample.	Invalid test.	Total Solids, CR4052-PD-1240	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test.
	4.1	Mercury	Damaged sample.	Invalid test.	Mercury, CR4052-PD-1300	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run test. Refer to Performance Evaluation CR4052-PD-0450
	4.1	Hexavalent Chromium	Damaged sample.	Invalid test.	Flame AA, CR4052-PD-1400	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test
	4.1	E-Coli - Wastewater	Damaged sample.	Invalid test.	E-coli in Wastewater, CR4052-PD-1500	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test
	4.1	HMX, RDX, TNT	Damaged sample.	Invalid test.	HMX, RDX, TNT, CR4052-PD-1600	Data Validation, CR4052-PD-0230	If data unacceptable, re-run test
	4.1	Nitrate-N	Damaged sample.	Invalid test.	Nitrate - N, CR4052-PD-1700	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run test Refer to Performance Evaluation CR4052-PD-0450
	4.1	Oil & Grease	Damaged sample.	Invalid test.	Oil and Grease, CR4052-PD-1800	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run test Refer to Performance Evaluation CR4052-PD-0450
	4.1	T. Cyanide	Damaged sample.	Invalid test.	Total Cyanide, CR4052-PD-1900	Performance Evaluations, CR4052-PD-0450 Data Validation, CR4052-PD-0230	If data unacceptable, re-run tes Refer to Performance Evaluation CR4052-PD-0450
5. Report Data	5.1	Write report	N/A	Inaccurate report	Customer Reports, CR4052-PD-0600	N/A	Revise/reissue report.
6. File Data	6.1	Distribute data to appropriate personnel and maintain copies as required.	N/A	N/A	Record Retention, CR4052-PD-0150	N/A	N/A

Note 1: All tests are consistent with EPA Methods as noted in the PD.

Document Control

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

March 28, 2002

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Jacquelyn J. Karty
Title	Quality Assurance Officer
Signature	Jacquelyn J. Karty

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NSWC REPOSITORY 08
3

I. SCOPE AND APPLICATION

This procedure describes the control of documents in Code 4052, including origination, review and revision, approval, use and control.

Documents utilized in Code 4052 will be maintained with current information and will be easily accessible.

II. SUMMARY OF METHOD

This method is consistent with ISO 9000 standard requirements.

III. QUALITY ASSURANCE

Quality is assured by distributing the responsibility for document control as follows:

Position	Responsibility
Branch Manager	Reviews and approve all changes in documents.
	Insures that all procedures are being followed.
Document Control Coordinator (DCC)	Maintains controlled electronic copies of all documents. Generates Master Document List upon request.
	Monitors document status to determine when review is required.
	Issues new controlled paper copies of procedures and attachments. Destroys old copies as appropriate.
Quality Assurance Officer (QAO)	Routes documents review and approval to the appropriate individuals.
	Provides periodic reports of document review status to Branch Manager.
	Issues procedure numbers for new documents.
	Assists others in the preparation of new procedures or modification of old procedures to ensure compliance with the Document Control Procedure and ISO Standards.
Laboratory Managers and Analysts	Reviews and creates documents as appropriate.
	Performs analysis and documentation according to documented procedures.
	Initiates document review as the procedures are modified.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Master Document List

The Master Document List is maintained as part of the electronic document repository system through Code 405. A current Master Document List can be generated by the DCC upon request.

Included in this procedure is a list of the location of controlled procedure copies, CR4052-PD-0100(1). For location of additional attachments, refer to CR4052-PD-0100(2). When a procedure is updated, the QAO provides a list to the DCC of appropriate places to send controlled copies.

B. Types of Procedures
Code 4052 operates using two types of procedures: Equipment Operating Procedures and Method Procedures.

1. Equipment Operating Procedures (EOP)
The EOP is specific to a piece or system of equipment. The intent of the procedure is to describe in general terms the theory and proper operation/maintenance of the particular piece of equipment. The EOP includes further reference to the appropriate Operation Manual provided by the manufacturer.

2. Method Procedures (PD)
The Method Procedure details the step-by-step application of the equipment and reagents in order to analyze a sample in a particular way. These procedures include sample preparation techniques, detailed equipment settings, and data analysis approaches for the methods routinely applied in the Code 4052 laboratory.

C. Common Formatting and Content

All EOP's and PD's are to have the following characteristics:

1. Cover Page
Each procedure includes a cover page to provide the title and approval information for the procedure. Refer to the cover page in this procedure for the proper format.

2. General Formatting
   a. Procedures are created using MS Word.
   b. Font: Ariel, 12pt.
   c. All margins: 1".
   d. Header ½" from the top.

e. Left justified.

D. Equipment Operating Procedure (EOP) Formatting and Content

1. EOP Body
EOP's include the following sections. If a section doesn't apply to the particular process, indicate "Not applicable" or "None", rather than omitting the section. Each section describes the type of information which may be appropriate to include, but does not limit the content of that section (note the capital letters in the section headers).

a. SCOPE AND APPLICATION: Identify the equipment to which the EOP is applicable. Include model numbers when available.

b. THEORY OF OPERATION: General description of the theory by which the equipment operates. This section includes basic principles and assessment of the equipment capability (describe the type of data the equipment generates).

c. FUNDAMENTAL OPERATION: Describe the general ways in which the equipment can be operated. Relate the equipment settings to the Theory of Operation.

d. PREVENTATIVE MAINTENANCE: Describe the required preventative maintenance recommended by the manufacturer or defined by operational experience. Define frequencies and material requirements of the maintenance. Refer to CR4052-PD-0200 as required.

e. CALIBRATION: Describe the required calibration recommended by the manufacturer or defined by operational experience. Define frequencies and material requirements of the calibration. Refer to CR4052-PD-0250 as required.

f. ATTACHMENTS: List all forms and attachments to the procedure. Each attachment is marked with procedure and attachment number, the revision number, and date (mm/dd/yy). Note the method in which the attachment is listed in this procedure.

Attaching uncontrolled reference documents is avoided. However, if necessary, these references can be attached if stamped "For Reference Only".

g. REFERENCES: List references that are not attached, but could be useful. Refer to the manufacturer's Operational Manual if available.

h. REVISION BLOCK: The revision table box includes revision number, date, description of the revision, and initials of the author of the revision. Refer to the revision block in this procedure for the appropriate format.

2. EOP Procedure Designation
Each procedure includes a header to list the procedure number, revision number, revision date, and page number of total pages excluding attachments. Refer to the header in this procedure for proper format (note 8pt Ariel font).

All Code 4052 methodology procedures are formatted as "CR4052-EOP-####".

E. Method Procedure (PD) Formatting and Content

1. PD Procedure Body
As in this procedure, documents include the following sections. If a section doesn't apply to the particular process, indicate "Not applicable" or "None", rather than omitting the section. Each section describes the type of information which may be appropriate to include, but does not limit the content of that section (note the capital letters in the section headers).

a. SCOPE AND APPLICATION: Describes the depth of the procedure, background discussion, and to whom the procedure applies.

b. SUMMARY OF METHOD: General description of the method and basis for the method, if applicable (such as EPA, Standard Methods, ISO Requirement).

c. QUALITY ASSURANCE: Describe any interferences that might be encountered in this method. Also highlight what steps this method incorporates for assuring quality. Examples may include the use of blanks, spikes, and raw samples. If this test is included in the performance evaluation process, refer to that procedure here.

d. APPARATUS: List of materials or equipment that may need to be gathered prior to beginning the procedure. Include any comments on special cleaning requirements (sterilization, for example).

e. SAMPLE HANDLING AND PRESERVATION: Includes containers, temperature, time, and preservation information for samples.

f. REAGENTS: List reagents required, preparation techniques, shelf life, etc.

g. PROCEDURE: Provide step-wise instructions designed for someone who is familiar with general laboratory work. Also include special clean-up requirements.

h. DATA ANALYSIS AND REPORTING: Describe the proper methods to handle data and refer to reporting requirements described in the reporting procedure.

i. ATTACHMENTS: List all forms and attachments to the procedure. Each attachment is marked with procedure and attachment number, the revision number, and date (mm/dd/yy). Note the method in which the attachment is listed in this procedure.

   Attaching uncontrolled reference documents is avoided. However, if necessary, these references can be attached if stamped "For Reference Only".

j. REFERENCES: List references that are not attached, but could be useful.

l. REVISION BLOCK: The revision table box includes revision number, date, description of the revision, and initials of the author of the revision. Refer to the revision block in this procedure for the appropriate format.

2. PD Procedure Designation
Each procedure includes a header to list the procedure number, revision number, date, and page number of total pages excluding attachments. Refer to the header in this procedure for proper format (note 8pt Ariel font).

The number scheme is as follows:

a. All Code 4052 methodology procedures are formatted as "CR4052-PD-####".
b. A 4-digit number is selected to group the procedure by the type of application:
   1) 0000 – 0699: Code 4052 Overall ISO System PD's
   2) 0700 – 0999: Code 4052 Overall Process PD's
   3) 1000 – 1999: NPDES Water Testing PD's
   4) 2000 – 2499: Drinking Water Testing PD's
   5) 2500 – 2999: Dye Project PD's
   6) 3000 – 3999: Sensitivity Testing PD's
   7) 4000 – 4999: Liquid Chromatography PD's
   8) 5000 – 5999: FBM Process PD's
   9) 6000 – 6999: Thermal Analysis Process PD's

C. Procedure Review Process

1. Frequency

All procedures will be reviewed as often as necessary to assure that they reflect current operating practices, including changes that result from changes in the method, technology, equipment, etc.

In addition, revisions may be initiated or suggested by any department personnel to improve instructions, correct mistakes, or modify operation requirements.

At minimum, each procedure will be reviewed every 4 years.

2. Process
a. When a review is required, the QAO will make sure an appropriate person is reviewing the procedure, providing assistance as required.
b. An uncontrolled, paper copy or electronic copy of the procedure can be requested from the DCC.

c. The marked-up copy or a corrected electronic copy will be given to the DCC to edit the controlled electronic file, issue new copies stamped "Controlled Copy" to the appropriate person as designated in CR4052-PD-0100(1,2). Controlled copies will be tracked using the receipts described in CR-405-3001.

d. Obsolete controlled documents will be returned to the DCC and retained according to procedure CR-405-3001.

3. Documenting Revision Content
Notes will be made in the revision block describing the nature of the revisions.

4. Approvals
Two approvals are required for each procedure. One approver must be the Branch Manager. The other approver must be a person who is familiar with the operation described in the procedure.

5. Training
Those reviewing the document will decide whether changes made were significant enough to warrant immediate training. Should training be warranted, the Branch Manager will be notified. Training will proceed as described in CR4052-PD-0300, Training.

D. Using the Procedures

1. Location of Controlled copies
Controlled paper copies will be distributed as described in CR4052-PD-0100(1). When possible, a copy will be located in the vicinity of the applicable process.

Controlled electronic copies will be maintained by the DCC in the document repository.

a. Document Receipt Log
Document Receipts (9ND-NWSCC 4430/10) will be controlled by the QAO and will be recorded on CR4052-PD-0100(3) Code 4052 Document Receipt Log.

It is the responsibility of the Receiving Person to ensure that the QAO is provided the receipt and to notify the QAO when the document is returned.

2. Making Copies

Copies of procedures cannot be made except by the DCC. These copies will be marked "Uncontrolled" and are not to be used for operation.

3. Marking-up Controlled Copies
The Quality Assurance Officer may make minor, temporary changes to a controlled copy of a procedure to ensure proper performance until the procedure can be revised. Any changes should be dated and initialed. Revisions should be complete within 3 months. It is not permissible for any other individuals to mark-up controlled copies.

E. Deleting a Procedure
Deleting a procedure will require the same level of approval as did the review or creation of a procedure. Simply mark "Deleted" in the revision block and handle as other revisions are handled. The document will be removed from the Master Document List.

VIII. DATA ANALYSIS AND REPORTING

A. Purpose

Test data is reported according to the operating procedure for the particular test. When appropriate, test logbooks (a logbook specific to a particular type of test) or instrument logbooks (a logbook for a particular instrument) are utilized to document activity.

B. Responsibility

It is the responsibility of the analyst to complete the appropriate information in the logbook. Notebooks should be maintained in a neat, orderly, and technically understandable manner. Ink should be used. Any errors made while entering data into the laboratory notebook will be crossed out with a single line, dated, and initialed.

C. Record Retention

Logbooks will be retained and located according to attachment CR4052-PD-0150(1). Removal of pages should be minimized; if it is necessary to remove a page(s), it should be noted in the logbook.

IX. ATTACHMENTS

CR4052-PD-0100(1) Code 4052 Master Document List, 10/01/2001

CR4052-PD-0100(2) Code 4052 Controlled Attachment Copy Inventory, 10/01/2001

CR4052-PD-0100(3) Code 4052 Document Receipt Log, 10/01/2001

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	11/1/00	Original issue.	MLS
1	3/28/01	Updated attachments.	MLS
2	10/01/2001	Added Document Receipt Log Requirements and Attachment and Changed header requirements and updated attachments.	CAW
3	3/28/2002	Updated Attachment CR4052-PD-0100(1)	CAW

Code 4052 Master Document List

PD Number	PD Title	Copy Locations (Note 1)
	4052 Overall Quality System PD's	
CR4052-PD-0001	Quality Assurance and Control Plan	C = Code 4052 QAO, Hunsicker
CR4052-PD-0100	Document Control	C = Code 4052 QAO, Hunsicker
CR4052-PD-0150	Record Retention	C = Code 4052 QAO, Hunsicker
CR4052-PD-0200	Preventative Maintenance	C = Code 4052 QAO, Hunsicker
CR4052-PD-0220	Chemical/Supply Management	C = Code 4052 QAO, Hunsicker
CR4052-PD-0230	Data Validation	C = Code 4052 QAO, Hunsicker
CR4052-PD-0250	Calibration	C = Code 4052 QAO, Hunsicker
CR4052-PD-0300	Training Overview	C = Code 4052 QAO, Hunsicker
CR4052-PD-0310	Training: Water Testing	C = Code 4052 QAO, Hunsicker
CR4052-PD-0450	Performance Evaluations	C = Code 4052 QAO, Hunsicker
CR4052-PD-0500	Chain of Custody: Soil and Water Samples	C = Code 4052 QAO, Hunsicker
CR4052-PD-0600	Customer Reports	C = Code 4052 QAO, Hunsicker
	4052 Overall Process PD's	
CR4052-PD-0700	Glassware Cleaning Procedure	C = Code 4052 QAO, Hunsicker
CR4052-PD-0710	Sterilization Procedure	C = Code 4052 QAO, Hunsicker
	NPDES Water Testing PD's	
CR4052-PD-1000	Metal Analysis Sample Preparation	C = Code 4052 QAO, Hunsicker
CR4052-PD-1100	Graphite Furnace	C = Code 4052 QAO, Hunsicker
CR4052-PD-1150	Flame AA Method	C = Code 4052 QAO, Hunsicker
CR4052-PD-1200	Arsenic/Selenium Sample Preparation & Analysis	C = Code 4052 QAO, Hunsicker
CR4052-PD-1210	TKN	C = Code 4052 QAO, Hunsicker
CR4052-PD-1220	Total Phosphorous	C = Code 4052 QAO, Hunsicker
CR4052-PD-1230	Ammonia-Nitrogen	C = Code 4052 QAO, Hunsicker
CR4052-PD-1240	Total Solids Determination	C = Code 4052 QAO, Hunsicker
CR4052-PD-1300	Mercury Sample Preparation & Analysis	C = Code 4052 QAO, Hunsicker
CR4052-PD-1400	Hexavalent Chromium Sample Preparation & Analysis	C = Code 4052 QAO, Hunsicker
CR4052-PD-1500	Escherichia Coli (E-Coli) in Wastewater	C = Code 4052 QAO, Hunsicker
CR4052-PD-1600	Pink Water Analysis	C = Code 4052 QAO, Hunsicker
CR4052-PD-1700	Nitrate - Nitrogen	C = Code 4052 QAO, Hunsicker
CR4052-PD-1800	Oil and Grease Sample Preparation & Analysis	C = Code 4052 QAO, Hunsicker
CR4052-PD-1900	Total Cyanide Preparation and Analysis	C = Code 4052 QAO, Hunsicker
CR4052-PD-2000	Total Coliforms and Eschericha Coli Analysis in Drinking Water	C = Code 4052 QAO, Hunsicker
	4052 FBM Process PD's	
CR4052-PD-5100	Cold Potting of Metallographic Test Specimens Using Epoxy Resins Araldite and Epoxicure	C = Code 4052 QAO
CR4052-PD-5200	Sample Order Maintenance in Lot Acceptance Testing of 95 and 150 grains per foot Flexible Linear Shape Charges (95 and 150 gr/ft FLSC)	C = Code 4052 QAO
CR4052-PD-5300	Sample Order Maintenance in Lot Acceptance Testing of 14.25 grains per foot Separation Linear Shape Charges (14.25 gr/ft SLSC)	C = Code 4052 QAO
	4052 Thermal Analysis Process PD's	
CR4052-PD-6000	Thermal Analysis Process Applications	C = Code 4052 QAO

Note 1: C=Controlled Copy, U=Uncontrolled Copy

Code 4052 Controlled Attachment Copy Inventory

Procedure Reference	Attachment Name	Location
Paper Copies		
CR4052-PD-0100(1)	Code 4052 Master Document List	QAO Office
CR4052-PD-0100(2)	Controlled Attachment Copy Inventory	QAO Office
CR4052-PD-0150(1)	Code 4052 Record Retention Detail	QAO Office
CR4052-PD-0200(2)	Water Systems Maintenance Log	B2707 Main Office Forms Drawer
CR4052-PD-0200(3)	Refrigerator/Freezer Temperature Log	B2707 Main Office Forms Drawer
CR4052-PD-0200(4)	Flame AA Maintenance Log	B2707 Main Office Forms Drawer
CR4052-PD-0200(5)	Code 4052 Maintenance Log	B2707 Main Office Forms Drawer
CR4052-PD-0220(1)	General Laboratory Supplies Inventory	B2707 Main Office Forms Drawer
CR4052-PD-0220(2)	NPDES Water Testing Supplies Inventory	B2707 Main Office Forms Drawer
CR4052-PD-0250(2)	Monthly Balance Verification	B2707 Main Office Forms Drawer
CR4052-PD-0300(1)	Code 4052 New Employee Orientation Training	B2707 Main Office Forms Drawer
CR4052-PD-0300(2)	Chemical Hygiene Plan Annual Review	B2707 Main Office Forms Drawer
CR4052-PD-0300(3)	Code 4052 Additional Training Form	B2707 Main Office Forms Drawer
CR4052-PD-0500(1)	Chain of Custody, NPDES Waste Water Samples	B2707 Main Office Forms Drawer
CR4052-PD-0500(2)	Chain of Custody, Sludge and Soil Samples	B2707 Main Office Forms Drawer
CR4052-PD-0500(3)	Code 4052 Internal Chain of Custody Form	B2707 Main Office Forms Drawer
CR4052-PD-0710(1)	Autoclave Use Report	B2707 Main Office Forms Drawer
CR4052-PD-1100(1)	Graphite Furnace Burn Log	B2707 Main Office Forms Drawer
CR4052-PD-1230(1)	Ammonia-Nitrogen Data	B2707 Main Office Forms Drawer
CR4052-PD-1400(1)	Hexavalent Chromium Data Analysis	B2707 Main Office Forms Drawer
CR4052-PD-1500(1)	E-Coli Analysis	B2707 Main Office Forms Drawer
CR4052-PD-1500(2)	Daily Temperature Record of Coliform Incubator	B2707 Main Office Forms Drawer
CR4052-PD-1700(1)	Nitrate-Nitrogen Analysis	B2707 Main Office Forms Drawer
CR4052-PD-1800(1)	Oil and Grease Analysis	B2707 Main Office Forms Drawer
CR4052-PD-1900(1)	Cyanide Data Sheet	B2707 Main Office Forms Drawer
Electronic Copies		
CR4052-PD-0600(1)	Sludge Testing Report	f:\Code4076\ISOForms
CR4052-PD-0600(2)	Water Testing Report	f:\Code4076\ISOForms
CR4052-PD-0600(3)	NPDES Report Cover Sheet	f:\Code4076\ISOForms
CR4052-PD-1150(1)	Water Analysis Metal Results Sheet	f:\Code4076\ISOForms
CR4052-PD-1150(2)	Sludge Analysis Metal Results Sheet	f:\Code4076\ISOForms
CR4052-PD-1200(1)	Arsenic in Sludge Analysis	f:\Code4076\ISOForms
CR4052-PD-1200(2)	Selenium in Sludge Analysis	f:\Code4076\ISOForms
CR4052-PD-1200(3)	Arsenic in Drinking Water Analysis	f:\Code4076\ISOForms
CR4052-PD-1200(4)	Selenium in Drinking Water Analysis	f:\Code4076\ISOForms
CR4052-PD-1210(1)	TKN Analysis	f:\Code4076\ISOForms
CR4052-PD-1220(1)	Total Phosphorous Analysis	f:\Code4076\ISOForms
CR4052-PD-1230(2)	Ammonia-Nitrogen Analysis	f:\Code4076\ISOForms
CR4052-PD-1300(1)	Mercury Data Analysis in Wastewater	f:\Code4076\ISOForms
CR4052-PD-1300(2)	Mercury Data Analysis in Sludge	f:\Code4076\ISOForms
CR4052-PD-1400(2)	Hexavalent Chromium Data Analysis	f:\Code4076\ISOForms
CR4052-PD-1900(2)	Total Cyanide Data Analysis	f:\Code4076\ISOForms

Code 4052 Document Receipt Log

Serial Number	Document Title or No.	Revision & Copy No.	Issuing Code	Date Received	Person Received	Date Returned

Record Retention

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

March 28, 2002

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Jacquelyn J. Karty
Title	Quality Assurance Officer
Signature	Jacquelyn J. Karty

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MDNC DEPOSITORY 08
CODE 405
COPY NO. 3

I. SCOPE AND APPLICATION

This procedure describes the retention of quality records created and/or filed in Code 4052. This includes but is not limited to test records, instrument records, instrument logbooks, and laboratory notebooks.

II. SUMMARY OF METHOD

This method is consistent with ISO standard requirements.

III. QUALITY ASSURANCE

All Code 4052 personnel are responsible for understanding and complying with the Code 4052 Record Retention procedure. The following personnel have specific responsibilities:

Position	Responsibility
Quality Assurance Officer (QAO)	Maintains controlled copies in forms drawer.
	Audit retention of documents and initiate proper disposal.
Laboratory Managers and Analysts	Copies blank forms from Forms Drawer.
	Complete and file forms, notebooks, records according to procedure.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Hard Copy Files

1. Hardcopy records may be filed in specified locations within Code 4052.
2. Record disposal will be via shredding when the retention time has been exceeded.
3. Records will be accessible to all individuals supporting the operation of the building.

B. Code 4052 Records, General

Quality Record	ISO 9000 Reference	Code 4052 Records	Record Retention Comments
Management Responsibility	4.1	Code 4052 Quality Assurance Summary Report	The Quality Assurance Summary Report is maintained by the Branch Manager. The current year plan and the previous year plan are retained at minimum.
Product ID and Traceability Inspection and Testing Control of Non-Conforming Product	4.8/4.10/ 4.13	Customer Reports (excluding NPDES)	Reports logged and retained per Naval Instructions.
Calibration	4.11	Internal Calibrations (Code 4052); External Calibrations (outside Code 4052)	Internal calibration records and environmental devices Calibration Reports will be retained per CR4052-PD-0150(1); External calibration records will be maintained by the calibrator.
Audit Results	4.17	External Audit Reports (outside 4052)	External Audit Reports are maintained by the Quality Assurance Officer for five years beyond the verification of all CARS.
Training Records	4.18	Training records for Code 4052 personnel	Records are retained per attachment CR4052-PD-0150(1); tests and additional training records are maintained by the Branch Manager for the period in which the employee is working in Code 4052.

C. Record Retention Detail

See attachment for detailed information concerning Code 4052 records not specifically described above.

D. Electronic Files

Document Control attachment CR4052-PD-0100(2) lists several documents maintained on the F:\ Code4076\ISOFORMS directory. The contents of this drive can be altered only by the Quality Assurance Officer. These documents are used to generate reports and analyze data. Results are printed and maintained per CR4052-PD-0150(1) and represent the official quality record. In addition, completed electronic forms are renamed and stored on the F:\Code4076\ISODATA subdirectory. Retention of these files allows for the efficient creation of corrected quality records if errors are discovered in the review process. THESE FILES DO NOT REPRESENT THE OFFICIAL QUALITY RECORDS. This directory will be maintained with no more than 5 years of data.

VIII. DATA ANALYSIS AND REPORTING

When contractually agreed upon, applicable test data is made available to the customer upon request.

IX. ATTACHMENTS

CR4052-PD-0150(1) Code 4052 Record Retention Detail, 3/28/01

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	11/1/00	Original issue.	MLS
1	2/8/01	Updated attachment 1. Added f:\code4076\ISODATA explanation.	MLS
2	6/06/01	Modified headers. Removed internal audit procedure reference	JJK
3	10/09/2001	Updated attachment 1. To include Certificates of Analysis in response to Audit Incident.	CAW
4	03/28/2002	Updated attachment 1. To include Calibration Reports for environmental devices.	CAW

Code 4052 Record F ion Detail

Area	Form or Record	Procedure Reference	Location of Form	Location of Record	Retention Time (Current Fiscal Year + ___)	Method of Indexing	Filing Responsibility
Overall System	Quarterly Quality Meeting Notes	CR4052-PD-0001	---	MA Office	3 years	Folder, Reverse-chronological	Management Assistant
	Monthly Routine Maintenance Log	CR4052-PD-0200(2)	B2707, Main Office	Water Lab	3 years	Folder, Reverse-chronological	Lab Analyst
	Weekly Controlled Temperature Log	CR4052-PD-0200(3)	B2707, Main Office	Water Lab	3 years	Binder; Folder, Reverse-chronological	Lab Analyst
	Flame AA Maintenance Log	CR4052-PD-0200(4)	B2707, Main Office	Water Lab	4 years	Folder, Reverse-chronological	Lab Analyst
	Semi-Annual Code 4052 Maintenance List	CR4052-PD-0200(5)	B2707, Main Office	Water Lab	5 years	Folder, Reverse-chronological	Lab Analyst
	Annual Code 4052 Maintenance List	CR4052-PD-0200(6)	B2707, Main Office	Water Lab	6 years	Folder, Reverse-chronological	Lab Analyst
	General Laboratory Supplies Inventory	CR4052-PD-0220(1)	B2707, Main Office	B2707, Main Office	1 year	Folder, Reverse-chronological	Lab Analyst
	NPDES Water Testing Supplies Inventory	CR4052-PD-0220(2)	B2707, Main Office	Water Lab	1 year	Folder, Reverse-chronological	Lab Analyst
	Data Validation Control Charts	CR4052-PD-0230	---	Water Lab	10 years	Binder, sorted by source	QAO
	Monthly Balance Verification	CR4052-PD-0250(2)	B2707, Main Office	Water Lab	3 years	Binder; Folder, Reverse-chronological	Lab Analyst
	Calibration Records	CR4052-PD-0250	---	Water Lab	5 years	Folder	Lab Analyst
	Chemical Hygiene Plan Annual Review	CR4052-PD-0300(2)	---	MA Office	3 years	Folder, Reverse-chronological	Management Assistant
	Code 4052 Additional Training Form	CR4052-PD-0300(3)	---	MA Office	3 years	Folder, Reverse-chronological	Management Assistant
	Code 4052 Job Traveler	CR4052-PD-0350(1)	B2707, Main Office	B2707, Main Office	3 years	Folder, Reverse-chronological	Program Manager
	Corrective Action Report Form	CR4052-PD-0400(1)	B2707, Main Office	B2707, Main Office	3 years	Active = Binder, Report number Complete=Folder, Report number	QAO
	Preventative Action Report Form	CR4052-PD-0400(2)	B2707, Main Office	B2707, Main Office	3 years	Complete=Folder, Report number	QAO
	Performance Evaluation Reports	CR4052-PD-0450	---	B2707, Main Office	5 years	Folder, Reverse-report number	QAO
	Chain of Custody: NPDES Water Samples	CR4052-PD-0500(1)	B2707, Main Office	Water Lab	5 years	Folder, Reverse-chronological	Lab Analyst
	Chain of Custody: Sludge and Soil Samples	CR4052-PD-0500(2)	B2707, Main Office	Water Lab	5 years	Folder, Reverse-chronological	Lab Analyst
	Code 4052 Internal Chain of Custody Form	CR4052-PD-0500(3)	B2707, Main Office	B2707, Main Office	5 years	Folder, Reverse-chronological	Lab Analyst
	Customer Reports (Data, Cover Sheet)	CR4052-PD-0600 (1-3)	F:\Drive	Water Lab	5 years	Binder, Reverse-chronological	Lab Manager
Overall Process	Autoclave Use Report	CR4052-PD-0710(1)	B2707, Main Office	Water Lab	2 years	Binder, Chronological	Lab Analyst
NPDES	Graphite Furnace Burn Log	CR4052-PD-1100(1)	B2707, Main Office	PW	3 years	Binder; Folder, Reverse-chronological	Lab Analyst
	GFAA Analysis Records	CR4052-PD-1100	---	Water Lab	3 years	Binder; Folder, Reverse-chronological	Lab Analyst
	Metal Analysis Log Book	CR4052-PD-1150	---	Water Lab	5 years	Cabinet	Lab Analyst
	Water Analysis Metal Results Sheet	CR4052-PD-1150(1)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	Sludge Analysis Metal Results Sheet	CR4052-PD-1150(2)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	Arsenic in Sludge Analysis	CR4052-PD-1200(1)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	Selenium in Sludge Analysis	CR4052-PD-1200(2)	F:\Drive	Water Lab	3 years	Folder, Reverse-chronological	Lab Analyst
	Arsenic in Drinking Water Analysis	CR4052-PD-1200(3)	F:\Drive	Water Lab	4 years	Folder, Reverse-chronological	Lab Analyst
	Selenium in Drinking Water Analysis	CR4052-PD-1200(4)	F:\Drive	Water Lab	5 years	Folder, Reverse-chronological	Lab Analyst
	TKN Analysis	CR4052-PD-1210(1)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	TKN Log Book	CR4052-PD-1210	---	Water Lab	5 years	Cabinet	Lab Analyst
	Total Phosphorous Analysis	CR4052-PD-1220(1)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	Total Phosphorous Log Book	CR4052-PD-1220(1)	---	Water Lab	5 years	Cabinet	Lab Analyst
	Ammonia-Nitrogen Data	CR4052-PD-1230(1)	B2707, Main Office	Water Lab	2 years	Spiral bound; chronological	Lab Analyst
	Ammonia-Nitrogen Analysis	CR4052-PD-1230(2)	F:\Drive	Water Lab	2 years	Binder, Reverse-chronological	Lab Analyst
	Sludge Log Book	CR4052-PD-1240	---	Water Lab	5 years	Cabinet	Lab Analyst
	Mercury Data Analysis in Wastewater	CR4052-PD-1300(1)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	Mercury Data Analysis in Sludge	CR4052-PD-1300(2)	F:\Drive	Water Lab	2 years	Folder, Reverse-chronological	Lab Analyst
	Hexavalent Chromium Analysis Data	CR4052-PD-1400(1)	B2707, Main Office	Water Lab	2 years	Spiral bound; chronological	Lab Analyst
	Hexavalent Chromium Data Analysis	CR4052-PD-1400(2)	F:\Drive	Water Lab	2 years	Binder, Reverse-chronological	Lab Analyst
	Media Log Book	CR4052-PD-1500	---	Water Lab	5 years	Cabinet	Lab Analyst
	E-Coli Analysis	CR4052-PD-1500 (1)	B2707, Main Office	Water Lab	2 years	Spiral bound; chronological	Lab Analyst
	Daily Temperature Record of Coliform Incubator	CR4052-PD-1500 (2)	B2707, Main Office	Water Lab	2 years	Binder; Chronological	Lab Analyst
	Explosive Water Log Book	CR4052-PD-1600	---	HPLC Lab	5 years	Cabinet	Lab Analyst
	Explosive Water HPLC Results	CR4052-PD-1600	---	Water Lab, HPLC Lab	2 years	Binder; Reverse-chronological	Lab Analyst
	Nitrate-Nitrogen Analysis	CR4052-PD-1700(1)	B2707, Main Office	Water Lab	2 years	Spiral bound; chronological	Lab Analyst
	Nitrate-Nitrogen LogBook	CR4052-PD-1700	---	HPLC Lab	5 years	Cabinet	Lab Analyst
	Nitrate-Nitrogen IC Results	CR4052-PD-1700	---	Water Lab	2 years	Binder; Reverse-chronological	Lab Analyst
	Oil and Grease Log Book	CR4052-PD-1800	---	Water Lab	5 years	Cabinet	Lab Analyst
	Oil and Grease Analysis	CR4052-PD-1800(1)	B2707, Main Office	Water Lab	2 years	Spiral bound; chronological	Lab Analyst
	Total Cyanide Logbook	CR4052-PD-1900	---	Water Lab	5 years	Cabinet	Lab Analyst
	Cyanide Data Sheet	CR4052-PD-1900(1)	B2707, Main Office	Water Lab	2 years	Spiral bound; chronological	Lab Analyst
	Total Cyanide Data Analysis	CR4052-PD-1900(2)	F:\Drive	Water Lab	2 years	Binder, Reverse-chronological	Lab Analyst
	Certificate of Analysis	---	B2707, Main Office	B2707, Main Office	Until use is completed	Cabinet	Lab Analyst
	C[illegible]ration Reports (environmental devices)	---	B2707, Main Office	B2707, Main Office	5 years	Cabinet	Lab Analyst

Preventative Maintenance

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

January 14, 2002

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Jacquelyn J. Karty
Title	Quality Assurance Officer
Signature	Jacquelyn J. Karty

MASTER

CONTROLLED COPY
NSWC REPOSITORY 03
CODE 405
COPY NO 3

I. SCOPE AND APPLICATION

This procedure describes the Explosive Sciences Branch Preventative Maintenance program.

II. SUMMARY OF METHOD

This method is consistent with ISO 9001 standard requirements.

III. QUALITY ASSURANCE

The laboratory personnel will conduct an orderly program of positive actions (equipment cleaning, reconditioning, adjusting, and/or testing) to prevent instruments or equipment from failure during use. The purpose of this preventative maintenance program is to increase measurement system reliability and thus data completeness and analysis efficiency.

Quality is assured by distributing the responsibility for preventative maintenance as follows:

Position	Responsibility
Branch Manager	Review PM progress in annual report.
Quality Assurance Officer (QAO)	Inspects maintenance log sheets for completeness.
Laboratory Managers and Analysts	Performs analysis and documentation according to documented procedures.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

Based on the purpose of each test, environmental influences, physical location of equipment, and manufacturer recommendations, a complete preventative maintenance plan has been developed, CR4052-PD-0200(1). The plan includes those steps completed when the equipment is used, steps completed when the equipment indicates that they are necessary, and steps taken on a routine basis.

A. CR4052-PD-0200(2) Water Systems Maintenance Log

This attachment is used to log the water conductivity for the water purification system (CR4052-IN-4000) and the automatic water still (CR4052-IN-4010).

B. CR4052-PD-0200(3) Controlled Temperature Log

This attachment is used to monitor temperature on a daily basis during the normal 5-day workweek.

C. CR4052-PD-0200(4) Flame AA Maintenance Log

Contains monthly and quarterly checklist for Flame AA Maintenance.

D. CR4052-PD-0200(5) Code 4052 Maintenance List

Includes miscellaneous tasks performed on a routine basis: graphite furnace lenses cleaning, UV-vis calibration, membrane replacement, various equipment cleaning, filter changes, and timer operation verification.

E. CR4052-PD-0200(6) Temperature Log for Oven Incubator

This attachment is used to record the temperatures for the oven incubator and water bath. The oven incubator has a thermometer on the top shelf and one on the bottom shelf. Both temperatures must be recorded 2 times per day. The reading must be at least 4 hours apart.

VIII. DATA ANALYSIS AND REPORTING

Maintenance records and status reports will be retained and located according to CR4052-PD-0150.

Any additional required maintenance beyond that described in CR4052-PD-0200(1) will be noted in the appropriate log books or data sheets.

IX. ATTACHMENTS

CR4052-PD-0200(1) Code 4052 Preventative Maintenance Summary

CR4052-PD-0200(2) Water Systems Maintenance Log

CR4052-PD-0200(3) Controlled Temperature Log

CR4052-PD-0200(4) Flame AA Maintenance Log

CR4052-PD-0200(5) Code 4052 Maintenance Log

CR4052-PD-0200(6) Temperature Log for Oven Incubator

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	12/1/00	Original issue.	MLS
1	5/02/01	Changed header requirements. Revised Section VII and attachments	JJK
2	10/19/01	Added CR4052-IN-4090 to attachments. Corrected references in CR4052-PD-0200(1).	JJK
3	01/14/02	Added CR4052-PD-0200(6) and CR4052-IN-4025.	JJK

Code 4052 Preventativ aintenance Summary

Code 4052 Instrument Number	Instrument Description	PM Each Time Used	Maintenance As Needed	Routine Maintenance
CR4052-IN-3020	ATOMIC ABSORPTION SPECTROMETER (GF/FLAME)	1. Verify adequate rinse water supply. 2. Verify drain is not full. 3. Check delivery tube alignment with graphite tube. 4. Calibrate with standards. 5. Record activity in log, CR4052-PD-1100(1).	1. Change graphite tube approximately every 200 runs. Check alignment after installation. Note in CR4052-PD-0200(2).	1. Clean lenses semiannually. Note in CR4052-PD-0200(5).
CR4052-IN-4020	REFRIGERATOR - FISHER	1. Read and log thermometer temperature readings, CR4052-PD-0200(3). 2. Check for spillage and wet areas around refridgerator.	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4025	REFRIGERATOR - ROPER	1. Read and log thermometer temperature readings, CR4052-PD-0200(3). 2. Check for spillage and wet areas around refridgerator.	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4100	HOOD - WATER LAB (1 OF 2)	N/A	1. Change bulbs as required.	N/A
CR4052-IN-4200	ASPIRATOR PUMP - WATER LAB (1 OF 2)	N/A	N/A	N/A
CR4052-IN-4230	OIL AND GREASE EXTRACTION	N/A	N/A	N/A
CR4052-IN-3000	ATOMIC ABSORPTION SPECTROMETER (FLAME)	1. Align lamps for optimum energy. 2. Align burner head for optimum absorbance. 3. Calibrate with standards.	N/A	1. Disassemble burner head, nebulizer and clean assembly with distilled water followed by acetone quarterly. Record in CR4052-PD-0200(4). 2. Clean optics quartely. Record in CR4052-PD-0200(4). 3. Check gaskets and o-rings quarterly. Record in CR4052-PD-0200(4). 4. Clean slot in burner monthly. After use, rinse burner and spray chamber with distilled water followed by acetone. Record in CR4052-PD-0200(4).
CR4052-IN-3010	As/Se HYDRIDE SYSTEM	1. Rinse with DI water after run complete. 2. Insert hydride solution for storage.	N/A	N/A
CR4052-IN-4250	EDL POWER SUPPLY	N/A	N/A	N/A
CR4052-IN-4260	EDL POWER SUPPLY II	N/A	N/A	N/A
CR4052-IN-4300	HOT PLATE/STIRRER	N/A	N/A	N/A
CR4052-IN-4150	pH METER	1. Calibrate meter with appropriate buffer solutions.	1. Replace electrode/probe and batteries.	NA

Code 4052 Preventativ[illegible] [illegible]intenance Summary

Code 4052 Instrument Number	Instrument Description	PM Each Time Used	Maintenance As Needed	Routine Maintenance
		2. Clean electrode after use and immerse in capped buffer reagent.		
CR4052-IN-4400	AUTOCLAVE	1. Fill reservoir. 2. Check for spillage and wet areas around drain. 3. Information recorded for each time cycle of operation in CR4052-PD-0710(1). 4. Sterility strips used with each cycle of operation. 5. Check drain screen, clean as necessary.		1. Automatic timer operation verified annually. Record in CR4052-PD-0710(1). 2. Clean with soap and water every 6 months. Record in CR4052-PD-0710(1).
CR4052-IN-4410	MICROPROCESSOR OVEN	1. Clean after each use if spills occur. 2. Set temperature settings and limits.	N/A	N/A
CR4052-IN-4500	BALANCE, TOP LOAD	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-4510	BALANCE, TOP LOAD	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-3030	MERCURY ANALYZER SYSTEM	1. Clean with water. 2. Check for leakage around tubing. 3. Calibrate high standard to 75% on chart recorder.	1. Clean with soap and water. 2. Replace Hg absorbing solution as needed.	N/A
CR4052-IN-4600	RECORDER	N/A	N/A	N/A
CR4052-IN-4420	OVEN	1. Clean after each use if spills occur. 2. Set temperature settings and limits.	N/A	N/A
CR4052-IN-4430	FURNACE	1. Check vent connection.	N/A	N/A
CR4052-IN-4610	DESCICATING CABINET	1. Verify N2 purge operational.	1. Change fittings as needed.	N/A
CR4052-IN-4620	DESCICATING CABINET	1. Verify N2 purge operational.	1. Change fittings as needed.	N/A
CR4052-IN-4520	BALANCE, ANALYTICAL	1. Clean after each use.	INACTIVE	INACTIVE
CR4052-IN-4700	COLIFORM INCUBATOR BATH	1. Check for spillage and wet areas around cover. 2. Ensure temperature setting is correct.	N/A	1. Verify thermostat setting with thermometer monthly when in use. Record in CR4052-PD-0200(3).
CR4052-IN-4710	COLONY COUNTER	N/A	1. Change bulbs as required.	N/A

Code 4052 Preventativ ıntenance Summary

Code 4052 Instrument Number	Instrument Description	PM Each Time Used	Maintenance As Needed	Routine Maintenance
CR4052-IN-4780	CULTURE INCUBATOR	1. Thermometer immersed in liquid and placed on shelf. 2. Temperature log twice daily during operation Apr - Nov per CR4052-PD-1500(2).	N/A	1. Clean with soap and water every annually. Record in CR4052-PD-0200(5).
CR4052-IN-3040	TOTAL ORGANIC CARBON ANALYZER	1. Check and or/replace pump tubing. 2. Prepare fresh reagent and standards. 3. Calibrate with standard.	1. Check reagent, waste, and gas flows. 2. Replace injection septum.	N/A
CR4052-IN-3050	UV/VIS SPECTROPHOTOMETER	1. Clean cells prior to and after each use. 2. Calibrate with standards.	N/A	1. Monitor lamp with didymium glass calibration filter every 6 months. Record in CR4052-PD-0200(5).
CR4052-IN-4730	MICROWAVE DIGESTION SYS.	1. Purge pressure monitoring line with 50 ml DI water. 2. Ensure exhaust fans (ICP, microwave) are on and working. 3. Ensure inside fan isn't touching vessels.	1. Check pressure monitoring tubing at vessel end.	1. Perform power calibration. Record in CR4052-PD-0250(3). 2. Clean with soap and water annually. Record in CR4052-PD-0200(5).
CR4052-IN-4110	HOOD - WATER LAB (2 OF 2)	N/A	1. Change bulbs as required.	N/A
CR4052-IN-4210	ASPIRATOR PUMP - WATER LAB (2 OF 2)	N/A	N/A	N/A
CR4052-IN-4740	CYANIDE DISTILLATION UNIT	1. Clean with water on external surfaces. 2. Check for leakage and ensure cold water head is snug in distillation tube. 3. Ensure heater in distillation housing unit is working.	N/A	1. Clean with soap and water annually. Record in CR4052-PD-0200(5). 2. Automatic timer operation verified annually. Record in CR4052-PD-0200(5).
CR4052-IN-4030	REFRIGERATOR - AVANTI	1. Read and log thermometer temperature readings, CR4052-PD-0200(3). 2. Check for spillage and wet areas around refridgerator.	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4750	WATER BATH	1. Check for spillage and wet areas around cover.	1. Clean with soap and water.	N/A
CR4052-IN-4800	THERMOMETERS	N/A	N/A	1. Externally calibrated.
CR4052-IN-4000	WATER PURIFICATION SYSTEM	1. Conductivity checked and logged monthly or when used in CR4052-PD-0200(2).	N/A	1. Change filters annually. Record in CR4052-PD-0200(5).
CR4052-IN-4760	FLASKSCRUBBER GLASSWARE WASHER	1. Wipe off soap dispenser.	N/A	N/A

Code 4052 Preventativ ıintenance Summary

Code 4052 Instrument Number	Instrument Description	PM Each Time Used	Maintenance As Needed	Routine Maintenance
CR4052-IN-4010	AUTOMATIC WATER STILL	1. Conductivity checked and logged monthly or when used in CR4052-PD-0200(2).	1. Filter cartridges replaced as needed.	N/A
CR4052-IN-4040	REFRIDGERATOR - LAB-LINE	1. Read and log thermometer temperature readings, CR4052-PD-0200(3). 2. Check for spillage and wet areas around refridgerator.	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4530	ANALYTICAL BALANCE	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-4160	ION ANALYZER	N/A	N/A	N/A
CR4052-IN-4170	AMMONIA SPECIFIC ION ELECTRODE	1. Calibrate meter with appropriate calibration solutions. 2. Clean electrode after use and immerse in capped buffer reagent. 3. Ensure slope is 60±3mV for ammonia per CR4052-PD-1230.	1. Replace electrode/probe as needed.	1. Every month, replace membrane and internal electrode solution. Record in CR4052-PD-0200(5).
CR4052-IN-2430 CR4052-IN-2140 CR4052-IN-21430 CR4052-IN-2150	AUTOSAMPLER CONDUCTIVITY DETECTOR LIQUID CHROMATOGRAPHY PUMP TEMP. CONTROL UNIT	1. Set column heater to 30°C. 2. Ensure rinse water bottle is full. 3. Calibrate with standards. 4. Check level mobile phase reservoir. Filter new mobile phase and purge reservoir line before use.	1. Repair/rebuild pumps, injection ports, install columns and seals.	N/A
CR4052-IN-4770	ULTRASONIC CLEANER	N/A	1. Change water as needed (DI water).	N/A
CR4052-IN-2060 CR4052-IN-2050	PHOTODIODE ARRAY DETECTOR SEPARATIONS MODULE	1. Calibrate with standards 2. Ensure adequate mobile phase. Fill reservoir before use if required. 1. Set column heater to 30°C.	1. Replace lamp 1. Repair/rebuild pumps, injection ports, install columns and seals as required. 2. Manufacturer representative for instrument PM service on internal components as requested.	1. Rebuild check valves, and replace pumps seals yearly.
CR4052-IN-4050	REFRIDGERATOR - SNAKE PIT	1. Read and log thermometer temperature readings, CR4052-PD-0200(3). 2. Check for spillage and wet areas around refridgerator.	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4310	HOT PLATE/STIRRER	N/A	N/A	N/A

Code 4052 Preventative Maintenance Summary

Code 4052 Instrument Number	Instrument Description	PM Each Time Used	Maintenance As Needed	Routine Maintenance
CR4052-IN-4630	DESCICATING CABINET	N/A	N/A	N/A
CR4052-IN-4220	ASPIRATOR PUMP	N/A	N/A	N/A
CR4052-IN-2010	MILLENNIUM CHROMATOGRAPHY SOFTWARE	N/A	N/A	1. Archive Projects monthly.
CR4052-IN-2030 CR4052-IN-2020 CR4052-IN-2040	PHOTODIODE ARRAY DETECTOR SEPARATIONS MODULE - 2690 TEMP. CONTROL UNIT	1. Calibrate with standards 2. Ensure adequate mobile phase. Fill reservoir before use if required. 3. Set column heater to 30°C.	1. Replace lamp 1. Repair/rebuild pumps, injection ports, install columns and seals as required. 2. Manufacturer representative for instrument PM service on internal components as requested.	1. Rebuild check valves, and replace pumps seals yearly.
CR4052-IN-2070	MILLENNIUM CHROMATOGRAPHY SOFTWARE	N/A	N/A	1. Archive Projects monthly.
CR4052-IN-2080 CR4052-IN-2100 CR4052-IN-2090 CR4052-IN-2110	AUTOSAMPLER MULTI-WAVELENGTH DETECTOR LIQUID CHROMATOGRAPHY PUMP TEMP. CONTROL UNIT	1. Ensure rinse bottle if full 2. Calibrate with standards 4. Check level mobile phase reservoir. Filter new mobile phase and purge reservoir line before use.	1. Repair/rebuild pumps, injection ports, install columns and seals. 2. Replace lamp.	1. Rebuild check valves, and replace pumps seals yearly.
CR4052-IN-2170	LIQUID CHROMATOGRAPHY PUMP	1. Check level mobile phase reservoir. Filter new mobile phase and purge reservoir line before use.		1. Rebuild check valves, and replace pumps seals yearly.
CR4052-IN-4060	REFRIGERATOR - GE	1. Read and log thermometer temperature readings, CR4052-PD-0200(3).	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4070	REFRIGERATOR - MAGIC CHEF	1. Read and log thermometer temperature readings, CR4052-PD-0200(3).	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4080	FREEZER - COOL-LAB	1. Read and log thermometer temperature readings, CR4052-PD-0200(3).	N/A	1. Annually clean with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4090	REFRIGERATOR/FREEZER-ROPER	1. Read and log thermometer temperature readings, CR4052-PD-0200(3).	N/A	1. Clean monthly with soap and water. Note in CR4052-PD-0200(5).
CR4052-IN-4540	ANALYTICAL BALANCE	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-4550	ANALYTICAL BALANCE	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.

Code 4052 Preventativ .intenance Summary

Code 4052 Instrument Number	Instrument Description	PM Each Time Used	Maintenance As Needed	Routine Maintenance
CR4052-IN-4560	ANALYTICAL BALANCE	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-4570	BALANCE, TOP LOAD	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-4580	ANALYTICAL BALANCE	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.
CR4052-IN-4590	ANALYTICAL BALANCE	1. Clean after each use.	1. Verify calibration with weights monthly per CR4052-PD-0250(2).	1. External certified calibration annually.

Water Systems Maintenance Log

Date	CR4052-IN-4000 Water Purification System Conductivity	CR4052-IN-4010 Automatic Water Still Conductivity	Initials

Controlled Temperature Log

Select	Equipment Number	Description	Temperature Range	Thermometer Serial Number	Correction Factor
	CR4052-IN-4020	Freezer	-20 - -10		
	CR4052-IN-4020	Refrigerator	1 – 5		
	CR4052-IN-4025	Freezer	-20 - -10		
	CR4052-IN-4025	Refrigerator	1 – 5		
	CR4052-IN-4030	Refrigerator	1 – 5		
	CR4052-IN-4040	Refrigerator	1 – 5		
	CR4052-IN-4050	Refrigerator	1 – 5		
	CR4052-IN-4060	Freezer	-20 - -10		
	CR4052-IN-4060	Refrigerator	1 – 5		
	CR4052-IN-4070	Refrigerator	1 – 5		
	CR4052-IN-4080	Freezer	-20 - -10		
	CR4052-IN-4090	Freezer	-20 - -10		
	CR4052-IN-4090	Refrigerator	1 – 5		
	CR4052-IN-4700	Water bath	44.5 ± 0.2		

Date	Reading[1] (°C)	Corrected Temperature[2] (°C)	Initials	Comments[3]

Note 1: Temperature as displayed on thermometer's stem.
Note 2: Temperature after adjustment by the thermometer's correction factor.
Note 3: Any corrective actions performed.

Flame AA Maintenance Log
CR4052-IN-3000

Monthly PM: Clean Burner Slot

Quarterly PM: Disassemble burner head, nebulizer and clean assembly with DI water, acetone
Clean optics.
Check gaskets and o-rings.

Date Competed	Check if Monthly PM	Check if Quarterly PM (Note 1)	Initials

Code 4052 Maintenance Log

Select	Equipment Number	Equipment Description	Maintenance Description	Frequency (Months)
	CR4052-IN-4170	Ammonia Ion Electrode	Replace membrane	1
	CR4052-IN-3020	Graphite Furnance	Clean lenses	6
	CR4052-IN-3050	UV-vis	Calibration	6
	CR4052-IN-4000	Water Purification System	Change Filters	12
	CR4052-IN-4400	Autoclave	Clean w/soap and water	6
			Verify proper timer operation	12
	CR4052-IN-4720	Culture Incubator	Clean w/soap and water	12
	CR4052-IN-4730	Microwave Digestion System	Clean w/soap and water	12
			Calibration	12
	CR4052-IN-4740	Cyanide Distillation Unit	Clean w/soap and water	12
	CR4052-IN-4020	Freezer	Defrost; clean w/soap and water	12
	CR4052-IN-4020	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4025	Freezer	Defrost; clean w/soap and water	12
	CR4052-IN-4025	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4030	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4040	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4050	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4060	Freezer	Defrost; clean w/soap and water	12
	CR4052-IN-4060	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4070	Refrigerator	Clean w/soap and water	1
	CR4052-IN-4080	Freezer	Defrost; clean w/soap and water	1
	CR4052-IN-4090	Freezer	Defrost; clean w/soap and water	12
	CR4052-IN-4090	Refrigerator	Clean w/soap and water	1

Date	Initials	Comments

Temperature Log for Oven Incubator

Thermometer	Serial Number	Correction Factor
Top Shelf		
Bottom Shelf		

Date	Time[1]	Top Shelf		Bottom Shelf		Initials	Comments
		Reading[2] (°C)	Corrected Temperature[3, 4] (°C)	Reading[2] (°C)	Corrected Temperature[3, 4] (°C)		

Note 1: The oven incubator temperature must be recorded 2 times per day. The readings must be at least 4 hours apart.
Note 2: Temperature as displayed on stem of thermometer.
Note 3: Temperature after adjustment by the thermometer's correction factor.
Note 4: Target Temperature range: 35 ± 0.5 °C

Chemical/Supply Management

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

December 1, 2000

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Louis C. Schwenk
Title	Laboratory Manager
Signature	Louis Schwenk

MASTER

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CODE 405 3

I. SCOPE AND APPLICATION

This procedure describes the process by which the laboratory ensures adequate quantity and quality of supplies for testing.

II. SUMMARY OF METHOD

This method is consistent with ISO 9001 standard requirements.

III. QUALITY ASSURANCE

A. Responsibility

Quality is assured by distributing the responsibility for document control as follows:

Position	Responsibility
Laboratory Manager	Performs routine supply audit.
	Orders supplies as required.
Laboratory Analyst	Labels and dates supplies appropriately.
	Notifies the laboratory manager of any additional supplies required.

B. Quality Practices

The following quality practices will be observed:

1. Standards to be used for calibration purposes are traceable to the National Institute of Standards and Technology (NIST).

2. Standard makeup solutions are prepared according to method specifications and documented in the specific analytical laboratory notebook with method of preparation, manufacturer, lot number, date of preparation, concentration, and analyst.

3. Purchased standards and laboratory standards will be stored in accordance with method or manufacturer requirements and recommendations.

4. The ESB shall label all reagents, solutions, and preservatives to indicate identity, concentration (where applicable), and the grade or quality of the material. Labeling shall also indicate the preparation or date the reagent was opened.

5. Outdated reagents and solutions will not be used unless their integrity is verified by testing.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Routine Inventory

The laboratory manager performs a routine inventory of supplies routinely consumed through testing.

1. General Lab Supplies

The general supplies required to operate the laboratory are listed in CR4052-PD-0220(1). This table indicates items required, suppliers, specification requirements, and minimum quantities. An inventory on these items is performed on a quarterly basis. Based on the current inventory and minimum quantity requirements, an appropriate order is placed and items in inventory beyond their shelf lives will be discarded appropriately.

Any receipts or documentation received in the ordering process is attached to the inventory sheet and filed according to Record Retention, CR4052-PD-0150.

2. NPDES Water Testing Supplies

The supplies required for NPDES water testing are listed in attachment CR4052-PD-0220(2). This table indicates items required and minimum quantities. An inventory on these items is performed on a monthly basis. Based on the current inventory and minimum quantity requirements, an appropriate order is placed and

items in inventory beyond their shelf lives will be discarded appropriately.

Any receipts or documentation received in the ordering process is attached to the inventory sheet and filed according to Record Retention, CR4052-PD-0150.

B. Additional Supplies Required

The laboratory manager is notified when it is observed that additional supplies are needed. The laboratory manager will either place an order at that time or wait until the next inventory period.

VIII. DATA ANALYSIS AND REPORTING

None.

IX. ATTACHMENTS

CR4052-PD-0220(1) General Laboratory Supplies, 12/1/00

CR4052-PD-0220(2) NPDES Water Testing Supplies, 12/1/00

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	12/1/00	Original issue.	MLS

General Laboratory Supplies

Inventory Completed by: _____ **Date:** _____

Item	Spec	Supplier	Minimum Quantity	Current Inventory	Amount Ordered
Chemicals					
Methanol	HPLC Grade	Fisher	4 x 4L		
Acetonitrile	HPLC Grade	Fisher	4 x 4L		
Supplies					
Chemwipes	NSN 7920-00-965-1709	Horizon Ind.	2 boxes		
Disposable Lab Coats, L	1209034	Safety & First Aid	1 box		
Disposable Lab Coats, XL	1209035	Safety & First Aid	1 box		
Disposable Lab Coats, 2XL	1209036	Safety & First Aid	1 box		
Disposable Lab Coats, 3XL	1209037	Safety & First Aid	1 box		
Disposable Lab Coats, 4XL	NS-4XL-SMS	Safety & First Aid	1 box		
Disposable Lab Coats, 5XL	NS-5XL-SMS	Safety & First Aid	1 box		
5cc disposable syringes	14-823-35	Fisher	2 boxes		
HPLC Columns	CP28079	Varian	2		
HPLC Guard Cartridges	CP28141	Varian	2		
Nitrile Gloves, XL	40101-350	VWR	2 boxes		
Nitrile Gloves, L	40101-348	VWR	2 boxes		
Nitrile Gloves, M	40101-346	VWR	2 boxes		
Syringe Filters, Nylon, PP Housing, 0.2 micron	26066	Restek	2 boxes		

NPDE[illegible] ter Testing Supplies

Item	Test	Minimum Quantity	Current Inventory	Amount Ordered
Chemicals				
Nitric Acid, Trace metal grade (Fisher Scientific)	Mercury, Metals	5, 5-pint bottles		
AA Standards, ~ 20 elements (Env. Res. Ass.)	Mercury, Metals	100 / 500 ml bottles		Reorder 2 months before expiration date
Stannous chloride - suitable for mercury analysis	Mercury, Metals	500 g		
Hydroxylamine hydrocholoride, 50% solution - suitable for mercury analysis	Mercury, Metals	500 ml		
Potassium permanganate, 5% solution - suitable for mercury analysis	Mercury, Metals	2 L		
Potassium persulfate, 5% solution - suitable for mercury analysis	Mercury, Metals	2 L		
1% $NH_4H_2PO_4$ in 1% HNO_3 matrix modifier - suitable for mercury analysis	Mercury, Metals	100 ml		
1% Palladium solution matrix modifier - suitable for mercury analysis	Mercury, Metals	100 ml		
1% $Mg(NO_3)_2$ solution matrix modifier - suitable for mercury analysis	Mercury, Metals	100 ml		
Sodium borohydride	As/Se	200 g		
Sodium iodide	As/Se	200 g		
Ammonium pyrrolidine dithiocarbamate (APDC)	Hexavalent chromium	20 g		
Yellow mercuric oxide	TKN	100 g		
H_2SO_4, Trace metal grade	TKN, Phosphorous, Mercury/metals	5, 5-pint bottles		
K_2SO_4	TKN	500 g		
NaOH	TKN, Total Cyanide, Phosphorous +	2 kg		
Ammonium chloride	TKN	500 g		
Sodium dihydrogenphosphate	Total Cyanide	1 kg		
KCn	Total Cyanide	100 g		
Silver nitrate, 0.0192N	Total Cyanide	2 L		
P-dimethyl-amino-benzalrhodonine	Total Cyanide	10 g		
Chloramine - T	Total Cyanide	100 g		
Barbituric acid	Total Cyanide	200 g		
Magnesium chloride powder (mg $Cl_2 \bullet 6\ H_2O$)	Total Cyanide	500 g		
Sulfamic acid	Total Cyanide	250 g		
Hydrochloric acid	As/Se	5, 5-pint bottles		
Sodium sulfate drying cartridges, 50/pack	Oil & Grease	2 packs		
Hexodecane	Oil & Grease	300 ml		
Stearic acid	Oil & Grease	500 g		
Hexane	Oil & Grease	2, 4-L bottles		

NPDES ter Testing Supplies

Item	Test	Minimum Quantity	Current Inventory	Amount Ordered
Acetone	Oil & Grease	2, 4-L bottles		
Antimony potassium tartrate	Phosphorous	100 g		
Ammonium molybdate	Phosphorous	250 g		
Ascorbic acid	Phosphorous	100 g		
Potassium dihydrogen phosphate	Phosphorous	100 g		
Ammonium persulfate	Phosphorous	100 g		
m-TEC agar	E-coli	250 g		
Urea	E-coli	250 g		
Supplies				
Extraction disks, 10/box	Oil & Grease	3 boxes		
0.45 micron 47mm cellulose esters membrane, 100/box		2 boxes		
Type A/E glass fiber filter, 47 mm, 100/box		2 boxes		
Sterile pipets, 5 ml		2 boxes		
Sterile pipets, 10 ml		2 boxes		
Sterile petri dishes, 55mm		2 boxes		
0.45 micron membrane filters, 47mm, gridded, sterile		2 boxes		
Absorbent pads, cellulose, 47mm		2 boxes		
Polycarbonate sample bottles, sterile, dechloromator added, 100 ml, 200 bottles/box		2 boxes		
Colilert, mo/mugg presence/absense reagent, 20 testers/box		8 boxes		
Colilert, presence/absense color comparator		2 comparitors		
PE known organisms, 3 coliform types				
PE coliform/E-coli sample sets		1 year		
Try ptic soy broth		500 g		
Autoclave sterilization strip indicator		1 box		
Graphite carbon tubes for graphite furnace, 25/box		1 box		

Inventory Completed by: _____

Date: _____

Data Validation

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

January 22, 2001

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Michelle L. Spellman
Title	Quality Assurance Officer
Signature	Michelle L. Spellman

MASTER

I. SCOPE AND APPLICATION

This procedure describes the Code 4052 data validation process.

II. SUMMARY OF METHOD

As performance data (% recovery, duplicate analysis, etc.) is collected, it is compiled in a software program to statistically characterize the analytical process in terms of percent relative standard deviation (%RSD).

Duplicate samples are used to monitor method precision; spiked percentage recovery data is used to monitor method accuracy.

III. QUALITY ASSURANCE

Quality is assured by distributing responsibility as follows:

Position	Responsibility
Quality Assurance Officer (QAO)	Enters data into program and interprets results.
Laboratory Managers and Analysts	Provides analytical process data.

IV. APPARATUS

The Code 4052 laboratory uses Windowchem™ Software's ControlChart! ™ software to generate quality control charts.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A separate control chart will be prepared for each concentration range represented in the outfall data.

Data Type	Source	Test	File Location
Spike Recovery	001	Silver	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\AG.ccp
		Cadmium	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\CD.ccp
		Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\CR.ccp
		Copper	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\CU.ccp
		Cyanide	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\CYAN.ccp
		Hex Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\HEXCR.ccp
		Mercury	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\HG.ccp
		Ammonia – N	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\NH3.ccp
		Nickel	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\NI.ccp
		Nitrate-N	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\No3spike.ccp
		Lead	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\PB.ccp
		Zinc	F:\CODE4076\CCPRO\ccpropls\qechart\001-spike\ZN.ccp
	101	Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\101-spike\O&G.ccp
	107	Nitrate-N	F:\CODE4076\CCPRO\ccpropls\qechart\107-spike\NO3.ccp
		Ammonia-N	F:\CODE4076\CCPRO\ccpropls\qechart\107-spike\NH3.ccp
		Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\107-spike\O&G.ccp
	301	Ammonia-N	F:\CODE4076\CCPRO\ccpropls\qechart\301-spike\NH3.ccp
		Nitrate-N	F:\CODE4076\CCPRO\ccpropls\qechart\301-spike\NO3.ccp
		Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\301-spike\O&G.ccp
	Exp.	HMX (107)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-spike\HMX_107.ccp
		RDX (107)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-spike\RDX_107.ccp
		TNT (107)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-spike\TNT_107.ccp
		HMX (301)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-spike\HMX_301.ccp
		RDX (301)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-spike\RDX_301.ccp
		TNT (301)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-spike\TNT_301.ccp
	OPR	Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\OPR-spike\O&G.ccp
	Metals	Silver Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\AG.ccp
		Cadmium Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\CD.ccp
		Chromium Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\CR.ccp

		Copper Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\CU.ccp
		Mercury Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\HG.ccp
		Nickel Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\NI.ccp
		Lead Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\PB.ccp
		Zinc Blank	F:\CODE4076\CCPRO\ccpropls\qechart\metalsblank-spike\ZN.ccp
Duplicates	001	Silver	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\AG.ccp
		Cadmium	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\CD.ccp
		Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\CR.ccp
		Copper	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\CU.ccp
		Cyanide	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\CYANIDE.ccp
		Hex Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\HEXCR.ccp
		Mercury	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\HG.ccp
		Ammonia – N	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\NH3.ccp
		Nickel	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\NI.ccp
		Lead	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicatefPB.ccp
		Zinc	F:\CODE4076\CCPRO\ccpropls\qechart\001-duplicate\ZN.ccp
	101	Cyanide	F:\CODE4076\CCPRO\ccpropls\qechart\101-duplicate\CYANIDE.ccp
		Hex. Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\101-duplicate\HEXCHROM.ccp
		Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\101-duplicate\O&G.ccp
	107	Nitrate – N	F:\CODE4076\CCPRO\ccpropls\qechart\107-duplicate\NO3.ccp
		Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\107-duplicate\O&G.ccp
	301	Ammonia – N	F:\CODE4076\CCPRO\ccpropls\qechart\301-duplicate\NH3.ccp
		Nitrate – N	F:\CODE4076\CCPRO\ccpropls\qechart\301-duplicate\NO3.ccp
		Oil & Grease	F:\CODE4076\CCPRO\ccpropls\qechart\301-duplicate\OG.ccp
	Exp.	HMX (107)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-duplicate\HMX_107.ccp
		RDX (107)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-duplicate\RDX_107.ccp
		TNT (107)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-duplicate\TNT_107.ccp
		HMX (301)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-duplicate\HMX_301.ccp
		RDX (301)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-duplicate\RDX_301.ccp
		TNT (301)	F:\CODE4076\CCPRO\ccpropls\qechart\exp-duplicate\TNT_301.ccp
PE Samples	3260	Silver	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Ag_qe.ccp
		Cadmium	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Cd_qe.ccp
		Cyanide	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Cn_qe.ccp
		Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Cr_qe.ccp

	Copper	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Cu_qe.ccp
	Mercury	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Hg_qe.ccp
	Ammonia – N	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Nh3_qe.ccp
	Nickel	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Ni_qe.ccp
	Nitrate – N	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\No3_qe.ccp
	Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\O&g_qe.ccp
	Lead	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Pb_qe.ccp
	Zinc	F:\CODE4076\CCPRO\ccpropls\qechart\3260QE\Zn_qe.ccp
WPO	Ammonia – N	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\NH3_QE.ccp
	Cadmium	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\CD_QE.ccp
	Chromium	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\CR_QE.ccp
	Copper	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\CU_QE.ccp
	Cyanide	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\CN_QE.ccp
	Lead	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\PB_QE.ccp
	Mercury	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\HG_QE.ccp
	Nickel	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\NI_QE.ccp
	Nitrate – N	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\NO3_QE.ccp
	Oil and Grease	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\O&G_QE.ccp
	Silver	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\AG_QE.ccp
	Zinc	F:\CODE4076\CCPRO\ccpropls\qechart\WPOQE\ZN_QE.ccp

VIII. DATA ANALYSIS AND REPORTING

Data will be entered into the appropriate program the day of analysis, before the samples are discarded.

A. Data Entry

1. Spike Analysis

Spike result, sample result, and amount of spike added are entered by the user. The percentage recovery is calculated and graphed. Standard deviations are calculated and graphed.

2. Duplicate Analysis

Multiple results are entered by the user. The program calculates %RSD and graphs appropriately, indicating standard deviation performance on the graph.

B. Data Interpretation

The QAO and lab analyst validate the quality of the duplicate and spiked sample data. If further analysis is required, it is then conducted and re-evaluated. Any unusual observations are recorded in the laboratory notebook.

IX. ATTACHMENTS

None.

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	1/22/01	Original issue.	MLS

Calibration

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

June 21, 2001

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J Maegerlein
Name	Jacquelyn Karty
Title	Chemist
Signature	Jacquelyn Karty

MASTER

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CODE 405
COPY NO. 3

I. SCOPE AND APPLICATION

This procedure describes the Explosive Sciences Branch Calibration program. The procedure includes a list of equipment used by the ESB and the process for calibrating the equipment.

II. SUMMARY OF METHOD

This method is consistent with ISO 9001 standard requirements.

III. QUALITY ASSURANCE

Appropriate calibration checks will be performed to verify the stability and validity of the sample and the performance of testing and analytical equipment.

Quality is assured by distributing the responsibility for calibration as follows:

Position	Responsibility
Branch Manager	Review calibration/verification progress in annual report.
Quality Assurance Officer (QAO)	Inspects calibration/verification log sheets for completeness.
Laboratory Managers and Analysts	Performs analysis and documentation according to documented procedures.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

Based on the purpose of each test, environmental influences, physical location of equipment, and manufacturer recommendations, a complete calibration program has been developed, CR4052-PD-0250(1). The program includes an equipment list and a description of the type of calibration performed. Some calibrations are performed as the analysis is completed, some calibration is done routinely by the laboratory, some are performed by external calibration personnel, and some calibration is supported by internal laboratory verification.

A. CR4052-PD-0250(2) Monthly Balance Verification

This attachment is used to verify performance on a monthly basis. An externally calibrated weight (calibrated annually) is placed on the balance and the result recorded. If the weight falls within the acceptable range, no further action is required. If the balance performance is not successfully verified, additional external calibration will be ordered and the equipment re-verified.

Verification can also be performed if it is suspected that the balance may not be performing correctly. Non-routine verification will be documented on the same attachment.

These balances are routinely calibrated externally on an annual basis

VIII. DATA ANALYSIS AND REPORTING

All external calibrations are documented and retained by the calibrator.

Internal calibrations and verifications are documented in the attachments to this procedure and are retained and located according to CR4052-PD-0150.

Any additional required calibration will be noted in the appropriate logbooks or data sheets.

IX. ATTACHMENTS

CR4052-PD-0250(1) NPDES Water Analysis Equipment

CR4052-PD-0250(2) Monthly Balance Verification

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	1/17/01	Original issue.	MLS
1	6/21/01	Updated attachments. Removed microwave power calibration.	JJK

NPDES Water / [illegible]s Equipment

Code 4052 Instrument Number	Crane Tag Number	Instrument Description	Purchased	Manufacturer	Model	Serial Number	Location	Calibration (Note 1)
CR4052-IN-3020		ATOMIC ABSORPTION SPECTROMETER (GF/FLAME)	1994	PERKIN ELMER	300/3300		PW	S
CR4052-IN-4020	No tag.	REFRIGERATOR - 1 OF 2	-	FISHER SCI.	-		Water Lab	NA
CR4052-IN-4100	No tag.	HOOD - 1 OF 2		FISHER SCI.	93-408Q	2704-4	Water Lab	NA
CR4052-IN-4200	No tag.	ASPIRATOR PUMP - 1 OF 2	.	BOEKEL	177000		Water Lab	NA
CR4052-IN-4230	No tag.	OIL AND GREASE EXTRACTION	1995	ENVIRONMENTAL EXPRESS	ENVIRON. STEP SAVER		Water Lab	NA
CR4052-IN-3000	00164053451000	ATOMIC ABSORPTION SPECTROMETER (FLAME)	1985	PERKIN ELMER	5000	128122	Water Lab	S
CR4052-IN-3010	No tag.	As/Se HYDRIDE SYSTEM	1990	PERKIN ELMER	MHS-10		Water Lab	S
CR4052-IN-4250	No tag.	EDL POWER SUPPLY					Water Lab	NA
CR4052-IN-4260	No tag.	EDL POWER SUPPLY II					Water Lab	NA
CR4052-IN-4300	Not listed on inventory: 164M008383	HOT PLATE/STIRRER	1980	LAB-LINE	NO. 1268	1093-4008	Water Lab	NA
CR4052-IN-4150	Not listed on inventory: 164M008384	pH METER	1986	ORION RESEARCH	EA920	TW38A	Water Lab	S
CR4052-IN-4400	No tag.	AUTOCLAVE	1999	MARKET FORGE	STERILMATIC		Water Lab	X
CR4052-IN-4410	10000029958000	MICROPROCESSOR OVEN	1987	LAB-LINE	3489M, IMPERIAL IV	02970358	Water Lab	NA
CR4052-IN-4500	No tag.	BALANCE, TOP LOAD	1999	METTLER	PB1501-SRS	1118461722	Water Lab	X/Lab
CR4052-IN-4510	No tag.	BALANCE, TOP LOAD	1981	OHAUS	GT4100		Water Lab	X/Lab
CR4052-IN-3030	10000111192000	MERCURY ANALYZER SYSTEM	1984	PERKIN ELMER	MAS 50A	57984	Water Lab	S
CR4052-IN-4600	164M028003	RECORDER	1998	THOMAS SCI.	SERVOGOR 102	AZ5101100000	Water Lab	NA
CR4052-IN-4420	Not listed on inventory: 60036000133000	OVEN		VWR SCI.			Water Lab	NA
CR4052-IN-4430	10000029962000	FURNACE	1987	THERMOLYNE	F30420C	41200516	Water Lab	NA
CR4052-IN-4610	Not listed on inventory: 164M018758	DESCICATING CABINET		FISHER SCI.			Water Lab	NA
CR4052-IN-4620	10000121371000	DESCICATING CABINET	1992	BOEKEL	---	---	Water Lab	NA
CR4052-IN-4520	10000029978000	BALANCE, ANALYTICAL	1987	OHAUS	G160R	001640	Storage	Inactive
CR4052-IN-4700	No tag.	COLIFORM INCUBATOR BATH	1999	PRECISION	MDL-251	699060329	Water Lab	NA
CR4052-IN-4710	No tag.	COLONY COUNTER		LEICA - QUEBEC DARKFIELD	3325	223451	Water Lab	NA
CR4052-IN-4720	164M028044	CULTURE INCUBATOR	1998	HACH	15E	0700498	Water Lab	NA

NPDES Water A[illegible]is Equipment

Code 4052 Instrument Number	Crane Tag Number	Instrument Description	Purchased	Manufacturer	Model	Serial Number	Location	Calibration (Note 1)
CR4052-IN-3040	00164050714000	TOTAL ORGANIC CARBON ANALYZER	1984	DOHRMANN	DC-80	HD1179	Water Lab	S
CR4052-IN-3050	00164026902000	UV/VIS SPECTROPHOTOMETER	1979	PERKIN ELMER	552	26842	Water Lab	S
CR4052-IN-4730	00164057957000	MICROWAVE DIGESTION SYS.	1989	CEM CORPORATION	MDS -2000	C50236	Water Lab	LAB
CR4052-IN-4110	No tag.	HOOD - 2 OF 2		FISHER SCI.	93-608Q	2704-5	Water Lab	NA
CR4052-IN-4210	No tag.	ASPIRATOR PUMP - 2 OF 2		BOEKEL	177000		Water Lab	NA
CR4052-IN-4740	164M010857	CYANIDE DISTILLATION UNIT	1996	ANDREWS GLASS CO.	MIDI-DIST		Water Lab	NA
CR4052-IN-4030	No tag.	REFRIGERATOR - 2 OF 2	-	AVANTI	-		Water Lab	NA
CR4052-IN-4750	No tag.	WATER BATH	1995	PRECISION	184	12Y6	Snake Pit	NA
CR4052-IN-4800	SC 91038	THERMOMETER: CR4052-IN-3300	---	---	---	9533	Snake Pit	X
CR4052-IN-4810	---	THERMOMETER: CR4052-IN-2300/2500	---	---	---	9110	Water Lab	X
CR4052-IN-4820	---	THERMOMETER: CR4052-IN-2300/2500	---	---	---	9510	Water Lab	X
CR4052-IN-4830	SC 91037	THERMOMETER: SPARE	---	---	---	64620	---	X
CR4052-IN-4840	OPL 91037	THERMOMETER: SPARE	---	---	---	---	---	X
CR4052-IN-4000	164M008376	WATER PURIFICATION SYSTEM	1989	MILLIPORE	---	10566C	Utility Lab	NA
CR4052-IN-4760	164M036412	FLASKSCRUBBER GLASSWARE WASHER	2000	LABCONCO	230V	000910953K	Utility Lab	NA
CR4052-IN-4010	164M003153	AUTOMATIC WATER STILL	1980	STOKES	171-2	78926	Utility Lab	NA
CR4052-IN-4040		REFRIDGERATOR					Utility Lab	NA
CR4052-IN-4530	10000111223000	ANALYTICAL BALANCE	1985	METTLER	AE160	D85657	IR LAB	X/Lab
CR4052-IN-4160	164M008389	ION ANALYZER		CORNING	250		Chromatography Lab	S
CR4052-IN-4170		AMMONIA SPECIFIC ION ELECTRODE		ORION			Chromatography Lab	S
CR4052-IN-2430	164M009621	AUTOSAMPLER		SHIMADZU	SIL-9A	80283DN	Chromatography Lab	S
CR4052-IN-2140	00164053476000	CONDUCTIVITY DETECTOR	1986	WATERS	430	430000659	Chromatography Lab	S
CR4052-IN-2170	164M009602	LC PUMP		WATERS	510		Chromatography Lab	S
CR4052-IN-2100	00164053476001	MULTI-WAVELENGTH DETECTOR	1986	WATERS	490	490001861	Chromatography Lab	S
CR4052-IN-2150	164M009615	TEMP. CONTROL UNIT	1990	WATERS	---	TCM-002038	Chromatography Lab	S

NPDES Water A[illegible]s Equipment

Code 4052 Instrument Number	Crane Tag Number	Instrument Description	Purchased	Manufacturer	Model	Serial Number	Location	Calibration (Note 1)
CR4052-IN-4770	10000050479000	ULTRASONIC CLEANER	1985	BRANSON	B-42	B104548	Chromatography Lab	NA
CR4052-IN-2050	164M036192	SEPARATIONS MODULE	2000	WATERS	2690 XE	G00SM4736M	Chromatography Lab	S
CR4052-IN-4050	1000001739000	REFRIDGERATOR					Snake Pit	NA
CR4052-IN-4310	10000109639000	HOT PLATE/STIRRER	1980	LAB-LINE	NO. 1268		Snake Pit	NA
CR4052-IN-4630	10000017478000	DESCICATING CABINET	1973	BOEKEL	121027	---	Snake Pit	NA
CR4052-IN-4220	No tag.	ASPIRATOR PUMP		BOEKEL	177000		Snake Pit	NA

Calibration Definitions: "Lab" - Laboratory personnel calibrated, "X" - External calibration performed, "S" - Calibrated with calibration standards, "NA" - Not applicable

Monthly Balance Verification

Select	Equipment Number	Crane Tag Number	Equipment Description	Location	Test Weight, g	Acceptable Range, g
	CR4052-IN-4500	SC 095347	Mettler Balance, Top Load	Water Lab	50	+/- 0.1
	CR4052-IN-4510	10000121369000 SC068835	Ohaus Balance, Top Load	Water Lab	50	+/- 0.1
	CR4052-IN-4520	10000029978000 SC057687	Ohaus Balance, Analytical	Storage	INACTIVE	INACTIVE
	CR4052-IN-4530	10000111223000 SC054068	Mettler Balance, Analytical	IR Lab	5	+/- 0.0003
	CR4052-IN-4540	10000111193000 SC051577	Mettler Balance, Analytical	Water Lab	5	+/- 0.0003
	CR4052-IN-4550	10000111224000 SC054211	Mettler Balance, Analytical	Thermal Lab	5	+/- 0.0003
	CR4052-IN-4560	00164050707000 SC049230	Mettler Balance, Analytical	Thermal Lab	INACTIVE	INACTIVE
	CR4052-IN-4570	SC 095346	Ohaus Balance, Top Load	IR Lab	50	+/- 0.1
	CR4052-IN-4580	1000002994300 SC057688	Ohaus Balance, Analytical	Chromatography Lab	5	+/- 0.0003
	CR4052-IN-4590	10000111225000 SC054067	Mettler Balance, Analytical	Chromatography Lab	5	+/- 0.0003

Date	Weight Set (SC #)	Weight, g	Initials	Comments

Training Overview

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

December 1, 2000

Approvals:

Name	Nancey Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Michelle Spellman
Title	Quality Assurance Officer
Signature	Michelle Spell

MASTER

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I. SCOPE AND APPLICATION

The purpose of this procedure is to define the training process for Code 4052 personnel.

II. SUMMARY OF METHOD

This method is consistent with ISO 9001 standard requirements.

III. QUALITY ASSURANCE

Quality is assured by distributing the responsibility for document control as follows:

Position	Responsibility
Branch Manager	Maintains personnel files containing training documentation.
	Qualifies employees.
Code 4052 Personnel	Administer training in areas of qualification, verifies training as required.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Training Overview

1. New Employee Orientation Training
Each new employee will receive orientation training. Refer to the attachment CR4052-PD-0300(1) for details. After the new employee has completed this initial training, job qualification training begins.

2. Annual Refresher Training
All employees must successfully participate in annual refresher training in the following areas:

a. Explosive Certification
The Branch Manager must observe the behavior and recommend each employee for continued explosive certification on an annual basis. A form is provided and retained by the safety department documenting that the individual has been recommended for certification on all of the procedures associated with explosives in Code 4052. The Branch Manager verifies continued knowledge through a passing test score on the Explosive Certification Training II Test and the Building Safety Orientation Test (refer to CR4052-PD-0300(1)).

b. Chemical Hygiene Plan
The Chemical Hygiene Plan is reviewed and discussed by the team on an annual basis. The Branch Manager orally questions to team to verify knowledge. Satisfied that the group understands the procedure, the Branch Manager qualifies the group as documented in CR4052-PD-0300(2).

3. Job Qualification: Water Testing
For employees involved in water testing, refer to CR4052-PD-0310. This procedure includes initial training and refresher training requirements.

B. Training Administration
All training will be administered by an individual qualified in that particular job.

C. Verification

1. Method of Verification

Job knowledge is verified by the training administrator in a manner appropriate for the particular segment of the training. The training checklists describe specific verification techniques.

If the trainee is unable to pass any portion of this training process, the Branch Manager will determine the appropriate course of action.

2. Documentation of Qualification

After the appropriate portions of the training documentation has been completed, it is reviewed and signed by the Branch Manager. At this point, the employee is qualified for that portion of their job.

D. Assessment of Training Needs

1. Job Qualification Training

The employee and the Branch Manager will determine which Job Qualification Training is appropriate.

2. Additional Training

Additional Training can come from any of the following sources:

a. Code 4052 personnel suggestions
b. Quality audits, Corrective Action Requests, Preventative Action Requests, etc.

Additional training is either documented in the Code 4052 Additional Training Form or in an individual's personal development plan as appropriate.

E. Retraining

Personnel off the job are retrained as determined on a case by case basis.

VIII. DATA ANALYSIS AND REPORTING

A. Record Retention

Training documentation and any written tests administered by Code 4052 in the orientation process will be retained per CR4052-PD-0150.

IX. ATTACHMENTS

CR4052-PD-0300(1) Code 4052 New Employee Orientation Training, 12/1/00

CR4052-PD-0300(2) Chemical Hygiene Plan Annual Review, 12/1/00

CR4052-PD-0300(3) Code 4052 Additional Training Form, 12/1/00

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	12/1/00	Original issue.	MLS

Code 4052 New Employee Orientation Training

Employee Name: _____

Training	Provided by:	Training Material	Verification Method	Verified by (2):	Date Completed
Explosive Certification Training I	Safety Department	Family Tests, Interactive Computer Test, Video	Passing Test Scores (1)		
Explosive Certification Training II	Code 4052	NAVSEAINST 8020.9A NAVSEA OP5, Chapter 3 Building Procedure CR4052-2707-0003A	80% Passing Test Score ("Qualification Evaluation for All Families of Explosive Materials")		
Building Safety Orientation	Code 4052	Ordnance Operations of Bldg. 143 CR4052-0143-001 Operations of Building 142 CR4051-142-0012 Standard Operation Procedure for the Demil Facility CR4052-DEMIL-0021	80% Passing Test Score ("Proficiency Evaluation of Explosive Safety Criteria in the Following Explosive Sciences Branch Personnel Occupied Facilities: Demil, Bldg. 143, and Bldg. 142")		
Materials Orientation	Code 4052	Chemical Hygiene Plan	Team Discussion		
FBM Orientation	Code 4052	FBMPI-2000 per D-4052-1002	80% Passing Test Score		
Department Quality Orientation	Code 4052	Quality Assurance, CR4052-PD-0001 Document Control, CR4052-PD-0100 Record Retention, CR4052-PD-0150 Preventative Maintenance, CR4052-PD-0200 Chemical Supply Management, CR4052-PD-0220 Corrective and Preventative Actions, CR4052-PD-0400 Chain of Custody, CR4052-PD-0500 Quality Assurance Plan, Code 405	80% Passing Test Score ("Code 4052 Quality Training")		

(1) Passing test score determined by department administering the test.
(2) Not required by training administered by outside department.

Qualification Certified by:	Date:

Chemical Hygine Plan Annual Review

Employee Name	Review Date

Qualification Certified by:	Date:

Code 4052 Additional Training Form

Description of Training:	
Reason for Training: (Audit #, CAR #, PAR #, etc.):	
Reference Number (SOP#, etc.):	
Date of Training:	
Training Administrator:	
Verification Method:	

Trainees:

Performance Evaluation

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

January 19, 2001

Approvals:

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Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Michelle L. Spellman
Title	Quality Assurance Officer
Signature	Michelle L. Spellman

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I. SCOPE AND APPLICATION

This procedure describes the Code 4052 Performance Evaluation program for NPDES Water Testing.

II. SUMMARY OF METHOD

Performance evaluation (PE) audits are routinely conducted to ensure data quality. PE audit results are used to evaluate laboratory technician, instrumentation, and analytical system performance.

The ESB laboratory participates in an annual blind Discharge Monitoring Report (DMR) submitted to the Federal EPA as well as quarterly double blind Proficiency Testing reported to Code 095.

This procedure also includes an internal blind testing program whereby samples of known composition are submitted internally to analysts for evaluation.

This method is consistent with ISO 9001 standard requirements.

III. QUALITY ASSURANCE

Quality is assured by distributing the responsibility for document control as follows:

Position	Responsibility
Branch Manager	Review results of performance evaluations testing.
Quality Assurance Officer (QAO)	Review results of performance evaluation testing. Issue CAR/PAR if appropriate.
	Generate and analyze quality control charts.
	Incorporates PE results in the Annual Quality Assurance Summary Report.
Laboratory Managers and Analysts	Performs analysis and documentation according to documented procedures.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Routine Tests Performed

The following table summarizes the routine performance testing content:

Test	Procedure	Annual DMR Blind	Quarterly PT Double Blind
Total Solids	CR4052-PD-1240	X	
Ammonia-Nitrogen	CR4052-PD-1230	X	X
Total Cyanide	CR4052-PD-1900	X	X
Oil and Grease	CR4052-PD-1800	X	X
Nitrate – Nitrogen	CR4052-PD-1700	X	X
Cadmium	CR4052-PD-1000/1100/1150	X	X
Copper	CR4052-PD-1000/1100/1150	X	X
Chromium	CR4052-PD-1000/1100/1150	X	X
Lead	CR4052-PD-1000/1100/1150	X	X
Nickel	CR4052-PD-1000/1100/1150	X	X
Silver	CR4052-PD-1000/1100/1150		X
Zinc	CR4052-PD-1000/1100/1150	X	X
Mercury	CR4052-PD-1300	X	X

B. Test Procedure: External performance samples

1. The ESB receives samples. These samples were prepared with a known composition and preserved by an outside laboratory.

2. The ESB analyzes the samples according to the proper procedures for the tests requested.

3. The ESB reports double blind results to customer per CR4052-PD-0600; blind DMR results are reported on the form included with the samples.

4. The customer submits the results to the outside laboratory.

5. The customer provides identification numbers of study and samples to the ESB.

5. The outside laboratory compares results to target values and the performance of other laboratories participating in the study. The outside laboratory then returns an assessment of the ESB data to the customer.

6. The customer sends a copy of the outside laboratory comparison to the ESB with a cover letter summarizing the performance and listing any actions requested.

7. Appropriate CARs will be issued, investigated and documented per CR4052-PD-0400.

C. Test Procedure: Internal performance samples

The ESB periodically has the need to conduct additional internal performance testing. The ESB may conduct internal performance testing for tests not covered in the external testing, certification of a new analyst, modification of a procedure or equipment, etc. The samples for these tests may be purchased or internally prepared. Samples are analyzed according to the appropriate procedures. If Corrective or Preventative action is necessary, CR4052-PD-0400 will then be followed.

VIII. DATA ANALYSIS AND REPORTING

All feedback from the customer and results from the outside laboratory is filed according to CR4052-PD-0150(1).

IX. ATTACHMENTS

None.

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	1/19/01	Original issue.	MLS

Chain of Custody: Soil and Water Samples

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

February 12, 2001

Approvals:

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Title	Chemist
Signature	Jacquelyn Karty

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I. SCOPE AND APPLICATION

Sample custody procedures are necessary to provide and maintain data validity and control. Samples must be under the control of the laboratory at all times after their transfer from the sample courier and during sample analysis. A chain-of-custody program provides legal support to the analytical results and tracking for each sample that is brought into the laboratory and during sample analysis.

This procedure describes the method for completing, maintaining, and storing the sample tracking mechanisms for soil and water samples received by the Explosive Sciences Branch (ESB).

II. SUMMARY OF METHOD

Proper sample tracking is accomplished with a Chain-of-Custody (CoC) form and the Inert Sample Log. The CoC form is generated in the field and used to record a sample's identity. The CoC shall accompany samples from the time of sampling until the time they are delivered and logged into the ESB electronic Inert Sample Log.

In addition, some customers require the use of an Internal Chain of Custody (ICoC) record to track a sample from delivery at the laboratory through preparation, analysis, and disposal.

This method is consistent with ISO 9000 standard requirements as well as state and federal testing regulations.

III. QUALITY ASSURANCE

Quality is assured by distributing the responsibility for chain of custody as follows:

Position	Responsibility
Quality Assurance Officer (QAO)	Accept and log samples.
Laboratory Managers and Analysts	Accept, log, analyze, and ship samples as required.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

A. General

The ESB laboratory will not be involved directly in sampling projects except in rare cases, in which at the time a sampling plan will be written. The laboratory may, however, be involved in assisting the client or others in designing and planning the sampling project. The laboratory will recommend such items as the proper sample containers and preservatives to be used, valid sample numbers, matrices and location, the use of background and composite samples, blanks, duplicates, appropriate parameters and analytical methods, sample packaging, handling, and shipment.

The main objective of any sampling effort is to obtain information that can be used to characterize the sample. It is imperative that the specific information desired is defined first. The ESB laboratory will allow for planning of the sampling project and reduce the possibility of wasted effort. If the information is to be used in a regulatory manner for program compliance, it is very important that the correct procedures are used for sampling and analysis. How the information will be used will dictate how to establish the specific project, sampling, analytical, and data objectives.

B. Sample Containers and Preservatives

The ESB laboratory will provide the necessary sample containers and preservatives upon request from any client. The containers provided by the laboratory will be obtained from an independent source. The containers will be provided to the client in the same condition as they were when supplied by the manufacturer. The containers and preservatives will comply with method requirements being utilized for the analysis.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. Receiving and Identifying Samples

All laboratory personnel that perform water sample analysis or the Quality Assurance Officer can accept samples delivered to the laboratory. These laboratory personnel will acknowledge the receipt of the sample by signing the Chain of Custody form. If a sample is brought in without a Chain of Custody form, one will be completed and signed by the courier and the laboratory personnel receiving the sample (CR4052-PD-0500(1), CR4052-PD-0500(2)).

The Chain of Custody form is used to record a sample's identity and provide a record showing custody from sampling to delivery. The Chain of Custody shall accompany samples from the time of sampling until the time they are delivered and logged into the ESB electronic Inert Sample Log.

Samples that cannot be related to an associated Chain of Custody because of inadequate, ambiguous, or nonexistent labeling, will be discarded unless the requestor is able to provide immediate identification. Only under those circumstances where sample identification is fairly obvious, will laboratory personnel make a special effort to identify and correlate unidentified samples.

B. Inspecting Samples

Upon receipt of the sample shipment and Chain of Custody forms, the receiving laboratory personnel will inspect all containers, sample documentation, sample identification, sample preservation, and holding time to verify sample integrity. Sample integrity refers to the cumulative end result of those factors which contribute to the overall validity of a sample. Sample integrity is promoted and preserved by adhering to adequate custodial, handling, and identification procedures by those individuals collecting the samples, up to the point of receipt of samples by the laboratory. When the samples are received for testing or analysis they are checked for:

1. Physical damage to samples because of inadequate packing and protection. Physical damage to the sample container may be the fault of the carrier due to abusive handling or may be the fault of the sender because of faulty packaging. If damage to the container is evident, the package will be carefully opened and its contents inspected. In the event of damage to the sample because of damage to the shipping container, the sender will be notified and the invalid sample discarded.

2. Loss of samples because of inadequate or improper sealing. This includes leakage of liquids from vials, loss of particulate material from filters or containers, inadequate sealing of solid absorbent sampling tubes, etc.

3. Contamination of samples due to inadequate separation of sample types or bulk sampling materials.

4. Improper use of special shipping procedures designed to preserve the samples at temperatures other than ambient. Most violations will result in the need to determine the loss of integrity and a decision regarding the disposition of the sample.

Any samples which do not meet this inspection will be rejected and/or the discrepancies noted on the CoC form and in the ESB Inert Sample Log. The receiving laboratory personnel will then sign the Chain of Custody and provide a copy of the Chain of Custody to the courier, if requested. The customer's point of contact will be immediately notified of the discrepancies.

C. Tracking Samples: Inert Sample Log

After the sample is identified and inspected by the ESB laboratory, the sample is entered into the Inert Sample Log, which is maintained on the branch file server and backed up daily. It is then properly stored until analysis is performed.

1. Open the file F:/Code4076/Samp_log/Inert00.xls. This is the Inert Sample Log.

2. Input information into file:

   a. Name of the person receiving the sample
   b. Date the sample was received
   c. Customer Name
   d. Sample identification and description
   e. Analysis requested
   f. Laboratory sample identification number
   g. Analyst assigned to the analysis

3. Save the file

4. After logging in the samples, the laboratory personnel will write the laboratory sample identification number on the sample container with permanent marker.

5. The laboratory personnel will then properly store the sample. The sample will remain secure until it is removed by the individual analyzing the sample.

6. The ESB laboratory will then file the original Chain of Custody form according to CR4052-PD-0150.

D. Additional Sample Tracking: Internal Chain of Custody

When required by the customer, an Internal Chain of Custody (ICoC) form is completed after the samples are entered into the inert sample log. This form will be kept near the place of storage for easy access during sample preparation and analysis. The ICoC is attached, CR4052-PD-0500(3).

Each time a sample is placed in or removed from the storage location, the movement is recorded on the ICoC.

When the ICoC sheet is full, the page number is filled out, a new sheet is initiated, and the full sheet is filed with the CoC forms.

When the project is completed, ICoC sheets are filed with the CoC forms.

E. Shipping Samples

Should sample shipment be required, the following guidelines will be considered:

1. Check the method to determine if packing and shipping instructions are included. If so, follow the Instructions given.

2. Divide samples into appropriate and compatible shipping groups. Liquids will be kept separate from other materials.

3. Select appropriate shipping container and packing material.

4. Make sure that the sample is properly and accurately identified in the Chain of Custody form and that all necessary paperwork accompanies the shipment.

F. Storage and Disposal

Upon completion of an analysis, samples will be stored and/or properly disposed of in accordance with local, state, and federal environmental laws.

VIII. DATA ANALYSIS AND REPORTING

Records are filed per CR4052-PD-0150(1).

IX. ATTACHMENTS

CR4052-PD-0500(1) NSWC, Crane Division, Chain of Custody Form, NPDES Waste Water Samples, 2/12/01

CR4052-PD-0500(2) NSWC, Crane Division, Chain of Custody Form, Sludge and Soil Sampling, 2/12/01

CR4052-PD-0500(3) Code 4052 Internal Chain of Custody Form, 2/12/01

X. REFERENCES

None.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	12/1/00	Original issue.	MLS
1	2/12/01	Updated attachments. Added internal chain of custody.	MLS

NSWC, C[illegible] Division
Chain of Custody Form: NPDES Waste Water Samples

ustomer Address:

OMMANDER
ODE 0953 BLDG 3260
AVSURFWARCENDIV
00 HIGHWAY 361, CRANE IN 47522

Report to:

Nann Stone
Phone No. (812) 854-3382
Fax No. (812) 854-4177

Control Number: _____

Parameters to be Tested	Preservative Used	Hold Time	Laboratory Sample No.	Name of Collector	Time of Collection	Sampling Date	GRAB = G COMP = C	Sample ID	Laboratory Sample No.	Name of Collector	Time of Collection	Sampling Date	GRAB = G COMP = C	Sample ID
H_3-(N)	H_2SO_4	28 Days												
admium	HNO_3	6 Mo.												
hromium	HNO_3	6 Mo.												
pper	HNO_3	6 Mo.												
ad	HNO_3	6 Mo.												
ckel	HNO_3	6 Mo.												
ver	HNO_3	6 Mo.												
nc	HNO_3	6 Mo.												
rcury	HNO_3	28 Days												
anide	NAOH	14 Days												
and Grease	H_2SO_4	28 Days												
O_3-N	Not Required	48 hours												
plosives NT, HMX, RDX)	Not Required	30 Days												
xavalent Chromium	Not Required	24 hours												
COLI 1 - 10/31 Only)	Not Required	6hours												

elinquished by:	Date/Time	Received by	Date/Time	COMMENTS:

NSWC, Crane Division
Chain of Custody Form: Sludge and Soil Sampling

Customer Address:

COMMANDER
CODE 0953 BLDG 3260
NAVSURFWARCENDIV
00 HIGHWAY 361, CRANE IN 47522

Report to:

Nann Stone
Phone No. (812) 854-3382
Fax No. (812) 854-4177

Control Number: _____

Parameters	Preservative Used	Hold Time	Laboratory Sample No.	Name of Collector	Time of Collection	Sampling Date	Sample ID	Plastic or Glass	Rush 0008AA	Rush 0008AB
Cadmium	HNO_3	6 mo.						Plastic		
Chromium	HNO_3	6 mo.						Plastic		
Copper	HNO_3	6 mo.						Plastic		
ead	HNO_3	6 mo.						Plastic		
ickel	HNO_3	6 mo.						Plastic		
ilver	HNO_3	6 mo.						Plastic		
inc	HNO_3	6 mo.						Plastic		
olybdenum	HNO_3	6 mo.						Plastic		
rsenic	HNO_3	6 mo.						Plastic		
elenium	HNO_3	6 mo.						Plastic		
lercury	H_2SO_4	14 Days						Plastic		
Total Solids	Not Required	7 Days								
otassium	HNO_3	6 mo.						Plastic		
O_3-N	Not Required	48 hrs.						Plastic		
KN	H_2SO_4	48 hrs.						Plastic		
hosphorous	H_2SO_4	28 Days						Plastic		
mmonia-N	H_2SO_4	28 Days						Plastic		

elinquished by:	Date/Time	Received by:	Date/Time

Code 4052 Internal Chain of Custody Form

Project Name:	Project Leader:	Customer
Job Order Number:	Date:	Customer POC
Charge Number:	Page: of	Address & Phone #

Sample ID	Date	Time	Reason for Handling

Customer Reports

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

September 13, 2001

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J Maegerlein
Name	Steve C. Weddle
Title	Quality Assurance Officer
Signature	Steve C Weddle

MASTER

CONTROLLED COPY
NSWC REPOSITORY 08
CODE 405
COPY NO 3

I. SCOPE AND APPLICATION

This procedure describes the Code 4052 customer data reporting process. Customer data reports are products.

II. SUMMARY OF METHOD

When a customer relationship is established, the requirements for the format and timing of data reporting is defined per CR4052-PD-0350. It is the intention of the ESB to report all data in the most timely, accurate, and understandable way possible.

This method is consistent with ISO 9000 standard and Navy reporting requirements.

III. QUALITY ASSURANCE

Quality is assured by distributing the responsibility for customer reporting as follows:

Position	Responsibility
Branch Manager	Reviews and approves all customer reports.
Quality Assurance Officer (QAO)	Reviews and approves customer reports for NPDES testing.
Laboratory Managers and Analysts	Generates, reviews, and issues customer reports.

IV. APPARATUS

Not applicable.

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

Not applicable.

VII. PROCEDURE

A. NPDES Reports

Specific procedures have been established for reporting NPDES data as described in this section.

1. Report Timing and Format

a. Routine NPDES Testing

1) Sludge Testing

Sludge samples are generally received at the beginning of the month. Data is reported to the customer per CR4052-PD-0600(1) as soon as possible.

2) Water Testing

Various water samples are received throughout a given month. The data is compiled and reported by the 10th of the month as stated in the Memorandum of Understanding, (dated 1/11/98, Reference Section) using CR4052-PD-0600(2). This report includes the following tests: Metals, Nitrate-Nitrogen, Ammonia-Nitrogen, Hexavalent Chromium, E-coli, Explosives, Oil and Grease, and Total Cyanide. The inert sample log is used as a checklist to make sure all data is reported.

Explosive water test results are verbally reported per CR4052-PD-1600.

b. Non-routine NPDES Testing

When non-routine NPDES tests are requested, results are included in the monthly report format. If the sample is designated Rush, results will be delivered in 5-7 days; Emergency samples will be reported as soon as possible.

2. Report Approval Process

a. Levels of Approval

Each report must be approved at three levels: Laboratory Manager, Quality Assurance Officer, and Branch Manager.

Each level of approval has the responsibility, upon discovery of a nonconformance, to return the report to the prior level for correction or further return back through the process. A CAR/PAR will be initiated where/when appropriate.

1) Laboratory Manager Approval

The Laboratory Manager review of the report includes but is not limited to following attributes:

a) Check the data reliability: review the logbooks and the spreadsheets to verify that each analysis was performed correctly and that the data is reasonable.

b) Check dates, sample designations, etc, with logbooks to ensure accuracy.

2) Quality Assurance Officer Approval

The Quality Assurance Officer review of the report includes but is not limited to the following attributes:

a) The data is validated per CR4052-PD-0230.

b) The QAO verifies that all forms have been filed out and retained completely.

3) Branch Manager

The Branch Manager review of the report includes but is not limited to the following attributes:

a) Analysis complete within appropriate hold times.

b) Results vs. Detection Limits: Make sure a number below detection limit is not inadvertently reported.

b. Cover Sheet

As each individual approves the report, a cover sheet is signed. The basic cover sheet is attached to this procedure, CR4052-PD-0600(3).

c. Issuing Report

After the report has been approved appropriately, the Laboratory Manager will issue the report to the customer.

B. General Reports

1. Report Formant

Reports generated by Code 4052 outside of the NPDES scope are created in the format given in attachment CR4052-PD-0600(4). This format is consistent with Navy requirements to ensure professional and precise reporting.

Overall formatting guidelines include:

a. The standard font for these reports is Courier 12.
b. Margins are 1" on all sides. The first page must have a 2" top margin to accommodate the letterhead.
c. There are 2 spaces after each period (sentences and numbers).
d. There is 1 space after a set of parentheses, "()".
e. Justification is to the left.
f. Abbreviations are avoided.
g. Enclosures are mentioned in order (for example, Enclosure 1 is mentioned in the body of the report before Enclosure 2).
h. Enclosures numbers are denoted at the bottom right corner.
i. File/codes typically found in reports include:

File/Code	Activity
8000/2	Off Station Correspondence
8000/3	Crane Army
8000/5	Code 09
8000/11	Code 40
8000/12	Code 60
8000/13	Code 70
8000/14	Code 80

j. Every new page of the report must have the subject line at the top of the page with 1 blank line separating the subject line from the report contents.
k. Tables should be centered in the page, when possible. One blank line should be left before and after the table.

l. Paragraphs in the body of the report are numbered according to CR4052-PD-0600(4).

2. Report Approval

After the report is completed, it is printed and approved by the Branch Manager. After the Branch Manager has approved and signed the original report, a date stamp is placed in the upper right side below the Ser number.

3. Report Distribution

a. When a report is completed, it is logged in the Branch logbook.

b. Prior to issuing the original report to the customer, three copies of the full report are made.

1) Author copy: Copy to be retained by the originator of the report.

2) Code 40211: A copy of the report is sent to status accounting.

3) Code 4052 Copy: A copy of the report is retained at the Branch.

c. A Green Board copy of the cover page is stamped with the circulation stamp at the bottom right corner, circulated, and filed according to Navy instructions.

VIII. DATA ANALYSIS AND REPORTING

NPDES completed reports/cover sheets will be stapled and filed together per CR4052-PD-0150(1).

Other reports will be handled according to Navy instructions.

IX. ATTACHMENTS

CR4052-PD-0600(1) Sludge Testing Report, 3/19/01

CR4052-PD-0600(2) Water Testing Report, 3/19/01

CR4052-PD-0600(3) NPDES Report Cover Sheet, 3/19/01

CR4052-PD-0600(4) Code 4052 General Report Format, 3/19/01

X. REFERENCES

Memorandum of Understanding between Code 40 and Public Works, 1/11/98.

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	1/19/01	Original issue.	MLS
1	3/19/01	Updated attachments. Added attachment (4) and general report formatting requirements.	MLS
2	9/13/01	Revised to identify reports as products and provide response to non-conformances discovered during approval process. Updated attachments to reflect revision and date.	CAW

EXPLOSIVES SCIENCE BRANCE COL J52 ANALYTICAL REPORT

DATE OF REPORT:	SAMPLING DATE:
SAMPLE ID:	RECEIPT DATE:

TO: NANNETTE STONE

SLUDGE ANALYSES

ANALYSIS	METHOD/ PROCEDURE	LAB NUMBER (N)	DATE ANALYZED	WET SLUDGE RESULT (NOTE 1)	UNIT	DRY SLUDGE RESULT	UNIT
CADMIUM	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
CHROMIUM	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
COPPER	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
LEAD	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
NICKEL	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
SILVER	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
ZINC	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
MOLYBDENUM	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L		MG/KG
ARSENIC	SM 3114B CR4052-PD-1200				MG/L		MG/KG
SELENIUM	SM 3114B CR4052-PD-1200				MG/L		MG/KG
MERCURY	EPA 245.1 CR4052-PD-1300				MG/L		MG/KG
% TOTAL SOLIDS	EPA 160.2 CR4052-PD-1240				%	—	—
POTASSIUM	EPA 3015 SW 846 , SM 311B CR4052-PD-1000 CR4052-PD-1150				MG/L	#DIV/0!	%
TOTAL NITROGEN (SUM OF TKN & TOTAL NO_3-N)	EPA 300.3, 351.4 CR4052-PD-1700, CR4052-PD-1210				MG/L	#DIV/0!	%
TOTAL PHOSPHORUS	EPA 365.2 CR4052-PD-1220				MG/L	#DIV/0!	%
NITRATE-NITROGEN	EPA 300.3 CR4052-PD-1700				MG/L	#DIV/0!	%
AMMONIA-NITROGEN	EPA 350.3 CR4052-PD-1230				MG/L	#DIV/0!	%

Note 1: B[illegible] upon 1kg/l density.

DRY SLUDGE MG/KG = MG[illegible] / (TOTAL SO[illegible].01)

DRY SLUDGE WT. PERCENT (%) = MG/L, NUTRIENT / (% TOTAL SOLIDS) X 100)

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

Explosive Analysis Results

Analyst:

Detectable Limit: 0.01 mg/l
Method Basis: EPA Method 8330
ESB Procedure: CR4052-PD-1600

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	HMX	RDX	TNT

Note: BDL = Below Detectable Limits

CR4052-PD-0600(2), Rev 2
9/13/01

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

Total Cyanide Results

Analyst:

Detectable Limit: 0.005 mg/l
Method Basis: EPA Method 335.3
ESB Procedure: CR4052-PD-1900

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	Total Cyanide, mg/l

Note: BDL = Below Detectable Limits

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

Oil and Grease Results

Analyst:

Detectable Limit: 1.4 mg/l
Method Basis: EPA Method 1664
ESB Procedure: CR4052-PD-1800

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	Oil and Grease, mg/l

Note: BDL = Below Detectable Limits

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

E-Coli Results

Analyst:

Detectable Limit:
Method Basis: EPA Method 1103.1
ESB Procedure: CR4052-PD-1500

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	Colony Count/100ml

Note: BDL = Below Detectable Limits

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

Nitrate-Nitrogen Results

Analyst:

Detectable Limit: 0.1 mg/l
Method Basis: EPA Method 300.0
ESB Procedure: CR4052-PD-1700

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	Nitrate-Nitrogen, mg/l

Note: BDL = Below Detectable Limits

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

Ammonia-Nitrogen Results

Analyst:

Detectable Limit: 0.03 mg/l
Method Basis: EPA Method 350.3
ESB Procedure: CR4052-PD-1230

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	Ammonia-Nitrogen, mg/l

Note: BDL = Below Detectable Limits

Explosive Science Branch Code 4052
Analytical Analysis

To: Nannette Stone
Date of Report:

Hex Chrome Results

Analyst:

Detectable Limit: 0.005 mg/l
Method Basis: EPA Method 218.4
ESB Procedure: CR4052-PD-1400

Sample ID	Lab ID Number	Date Sampled	Date Received	Date Analyzed	Hex Chrome, mg/l

Note: BDL = Below Detectable Limits

From: Code 4052 (Louis Schwenk)
Phone: 854-5511 Fax: 854-5054

To: Code 095 (Nannette Stone)
Phone: 854-3382 Fax: 854-4177

Date: _____

Subject: NPDES AND OTHER PUBLIC WORKS SAMPLES ANALYSIS RESULTS FOR THE MONTH OF _____.

1. As requested in the Memorandum of Understanding (MOU) between Code 40 and Code 09 analysis data results are being forwarded to Code 095 by means of this facsimile.

2. Data included:

Data	Included
Total solids	
Potassium	
Nitrate-nitrogen	
Total cyanide	
Oil and Grease	
Ammonia-nitrogen	
Total kjeldahl nitrogen	
Total phosphorous	
Hexavalent chromium	
Metals	
E-coli	
Explosives	

REPORT REVIEWED BY:	DATE:
REPORT REVIEWED BY: QA/QC OFFICER	DATE:
REPORT REVIEWED BY:	DATE:

Code 4052 General Report Format

file code 8000/?
our code/report s/n Ser 4052/????
Refer to CR4052-PD-0600 for code listing. The 2nd line must be flush right and the 1st line aligned directly above line 2.
MEMORANDUM *(ALL CAPS) There are 2 blank lines between Ser line and MEMORANDUM line. 1 blank line after MEMORANDUM line.*
From: 4052 *2 blank spaces after colon.*
To: Name *Everything between here and the body of the report*
Address *is aligned directly under code above.*
1 blank line to separate address from subject line.
Subj: SUBJECT LINE MUST BE ALL CAPS
1 Blank line to separate subject line from references.
Ref: (a) First reference
(b) Second reference
1 blank line to separate references from enclosures.
Encl: (1) First enclosure *Confirm that this title matches*
(2) Second enclosure *title on the enclosure.*
1 blank line to separate enclosures from body of the report.
1. Background *Indent the paragraph line with 4 spaces.*
1 blank line to separate paragraph title and data.
a. Sub paragraphs are indented 4 additional spaces and identified with a small case letter. The second line of subparagraphs will continue to the left margin without indentation.
b. If subparagraphs are needed, use at least two.
1 blank line to separate report information from next paragraph.
2. Introduction
1 blank line to separate paragraph title and data.
Report information here.
1 blank line to separate report information from next paragraph.
3. Analytical Test Procedures
1 blank line to separate paragraph title and data.
Report information here.
1 blank line to separate report information from next paragraph.
4. Results and Discussion
1 blank line to separate paragraph title and data.
Report information here.
1 blank line to separate report information from next paragraph.
5. Closing (POC Paragraph)
1 blank line to separate paragraph title and data.
The closing paragraph must contain the POC, their code, and their extension. Never let proper names continue onto two lines.
4 Blank lines here

NANCEY J. MAEGERLEIN *Name is in all caps*

Glassware Cleaning Procedure

Explosive Sciences Branch
Naval Surface Warfare Center Crane
300 Highway 361
Crane, IN 47522

January 1, 2001

Approvals:

Name	Nancey J. Maegerlein
Title	Branch Manager
Signature	Nancey J. Maegerlein
Name	Jacquelyn J. Karty
Title	Chemist
Signature	Jacquelyn J. Karty

MASTER

CONTROLLED COPY
NSWC REPOSITORY 08
CODE 405
COPY NO. 3

I. SCOPE AND APPLICATION

This document provides the proper cleaning procedure of all glassware in the Explosive Science Branch, Code 4052.

II. SUMMARY OF METHOD

These procedures describe the method for removing surface residuals, washing and drying, and storage of the glassware.

III. QUALITY ASSURANCE

Not applicable.

IV. APPARATUS

The following pieces of apparatus are used in this analysis:

- Flask Scrubber Glassware Washer, Model 230V

V. SAMPLE HANDLING AND PRESERVATION

Not applicable.

VI. REAGENTS

The following reagents are required in this analysis.

A. Dish detergent, concentrated low foaming cleaning agent.

VII. PROCEDURE

A. Rinsing Glassware After Use

Glassware is rinsed after use to minimize the contamination of the dishwasher and the possibility of personnel exposure.

1. Dye-Contaminated Glassware: Rinse 3 times with methanol.

2. Organic-Contaminated Glassware: Rinse with tap water.

B. Removing Surface Residuals

Any tape, label or marker stains must be removed prior to placing in the dishwasher. Tape and labels may be removed with a knife, while marker stains can be removed with acetone.

C. Washing/Drying the Glassware

1. Place all glassware onto steel or plastic-coated racks.

2. Add soap to the dishwasher, both sides of the soap compartment.

3. Close and lock the door.

4. Scroll through programs using the "Select Program" button. Select the "Glass Plus High Heat" cycle.

5. Verify that the "Pure" button has been selected to use DI water rinse.

6. Press "Start" to run the dishwasher.

7. Turn sign to "Clean".

D. Removal and Storage of Glassware

1. After the cycle is complete, unload the dishwasher onto the transporting rack.

2. Turn sign to "Dirty"

3. Return clean glassware to storage cabinets in the appropriate sections or to the appropriate segregation tubs (Oil and Grease analysis, Hexavalent chromium analysis, and Cyanide analysis).

   Note: Store volumetric flasks with caps in place to prevent contamination.

E. Rinsing the Glassware Prior to Use

All glassware should be rinsed three times with the solvent to be used in the particular analysis to be performed.

VIII. DATA ANALYSIS AND REPORTING

None.

IX. ATTACHMENTS

None.

X. REFERENCES

Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples.

Flask Scrubber Glassware Washer, Model 230V Operations Manual

XI. REVISION BLOCK

REV	DATE	REVISION	AUTHOR
0	1/1/01	Original issue.	MLS

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

James M. Hussiter
SIGNATURE

Environmental Protection Department Manager 8/01/02
TITLE DATE