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FINAL SAMPLING AND ANALYSIS PLAN FULL RESOURCE CONSERVATION AND RECOVERY ACT FACILITY INVESTIGATION SOLD WASTE MANAGEMENT UNIT 60 FORMER LANDFILL AT THE MARINA WITH TRANSMITTAL AND RESPONSE TO COMMENTS NAVAL ACTIVITY PUERTO RICO
6/7/2012
BAKER ENVIRONMENTAL, INC.

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June 7, 2012

U.S. Environmental Protection Agency - Region II
290 Broadway – 22nd Floor
New York, New York 10007-1866

Attn: Mr. Adolph Everett, P.E.
Chief, RCRA Programs Branch

Re: Contract N62470-10-D-3000
IDIQ for A/E Services for Multi-Media
Environmental Compliance Engineering Support
Delivery Order (DO) JM07
U.S. Naval Activity Puerto Rico (NAPR)
EPA I.D. No. PR2170027203
Final Sampling and Analysis Plan Full RCRA Facility Investigation SWMU 60

Dear Mr. Everett:

Michael Baker Jr., Inc. (Baker), on behalf of the Navy, is pleased to provide you with one hard copy and one electronic copy provided on CD of the replacement pages that make up the Final Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) Full RCRA Facility Investigation SWMU 60 – Former Landfill at the Marina. Directions for inserting these pages into the Draft Sampling and Analysis Plan Full RCRA Facility Investigation Report for SWMU 60 are enclosed.

This report is being submitted in accordance with EPA comments dated April 26, 2012. The Navy responses to these comments are attached for your review. Additional distribution has been made as indicated below.

If you have questions regarding this submittal, please contact Mr. Mark E. Davidson at (843) 743-2124.

Sincerely,
MICHAEL BAKER JR., INC.

Mark E. Kimes, P.E.
Activity Coordinator

Attachments

cc: Ms. Debra Evans-Ripley, BRAC PMO SE (letter only)
Mr. David Criswell, BRAC PMO SE (letter only)
Mr. Tim Gordon, US EPA Region II (1 hard copy and 1 CD)
Mr. Mark E. Davidson, BRAC PMO SE (1 hard copy and 1 CD)
Mr. Stacin Martin, NAVFAC Atlantic (1 CD)
Mr. Pedro Ruiz, NAPR (1 electronic copy only)
Mr. Jose Font, US EPA Caribbean Office (1 hard copy and 1 CD)
Ms. Bonnie Capito, NAVFAC Atlantic – Code EV42 (1 hard copy)
Mr. Gloria Toro, PR EQB (1 hard copy and 1 CD)
Ms. Wilmarie Rivera, PREQB (1CD)
Mr. Felix Lopez, US F&WS (1CD)
Ms. Connie Crossley, Booz Allen Hamilton (1 CD)

NAVY RESPONSE TO EPA COMMENT LETTER DATED APRIL 26, 2012
ON THE
DRAFT SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
FOR SWMU 60 DATED DECEMBER 30, 2011

The following comments were generated based on review of the *Draft Sampling and Analysis Plan (Field Sampling Plan and Quality Assurance Project Plan) for SWMU 60* (December 30, 2011) located at Naval Activity Puerto Rico (NAPR), Ceiba, Puerto Rico. EPA and PREQB comments are provided in italics, and the Navy's responses are provided in regular print.

EPA COMMENTS

GENERAL COMMENTS

EPA General Comment 1: *Throughout the SAP there are references to previous site characterizations conducted at SWMU 60 between 1999 and 2004, and a Phase I Resource Conservation and Recovery Act (RCRA) Facility Investigation (Phase I RFI) conducted in January 2009; but relevant excerpts from these documents (i.e., previous sampling results and conclusions) are not included in the SAP to support the rationale for the proposed sampling. The Uniform Federal Policy on Quality Assurance Project Plans Guidance (UFP QAPP Manual), dated March 2005 indicates that each reference to a previous document should include a full reference that cites the year, location of the referenced document (appendix/attachment), and the page number of the reference. Revise all references in the SAP to previous site characterizations conducted in 1999, 2004, and the 2009 Phase I RFI to include this information. Ensure that the information provided includes data and conclusions to support the sampling rationale presented in the SAP.*

Navy Response to EPA General Comment 1: The reference requirements cited in the UFP QAPP Manual are geared towards instances where some or all of the required SAP element groups are incorporated into other project planning documents such as field sampling plans, field operations plans, project operations plans, or general project work plans. Relevant historical data and conclusions from the 1999 Site Characterization, 2004 ECP Investigation, and 2009 Phase I RFI are discussed in Worksheet #10 (Conceptual Site Model) and support the rationale for the proposed sampling presented in this SAP. Furthermore, analytical data exceeding applicable screening values from the 2009 Phase I RFI (investigation that provides the primary basis for this SAP) are shown on Figures 5 through 9. Existing data gaps and the data required to address those data gaps are clearly identified in Worksheet #11 (Project Quality Objectives/Systematic Planning Process Statements). References to these previous investigations are presented in the "References" section of this SAP as well as in Worksheet #13 (Secondary Data Criteria and Limitations Table). No revisions to the SAP area warranted.

EPA General Comment 2: *The SAP is missing information regarding decision criteria and rationale for permanent monitoring well (MW) placement at SWMU 60. It is unclear if the new MWs will be installed in upgradient/downgradient areas with known detections of Total Petroleum Hydrocarbons (TPHs), polycyclic aromatic hydrocarbons (PAHs), and metals. It is also unclear if the data is intended to be used to substantiate/compliment previously collected data, even if only screening data. In order to make informed guided decisions and to substantiate the proposed approach for the Full RFI, a clear rationale for permanent MW placement should be included in the SAP.*

Navy Response to EPA General Comment 2: Section 17.3.1 of Worksheet #17 (Sampling Design and Rationale) states that the monitoring wells (except 60SB13) will be located within and downgradient of the suspected debris disposal areas to characterize worst-case groundwater conditions and delineate the

extent of contamination. As agreed to during the project scoping session on September 28 and 29, 2011 (see Worksheet #9), the specific number and locations of these wells will be based on results of the geophysical survey described in Section 17.2.1 and contingent upon approval from the Navy, USEPA, and PREQB. Well 60SB13 will be located in the presumed downgradient direction of the former underground piping system associated with the marina's ASTs. No revisions to the SAP are warranted.

EPA General Comment 3: *Neither Worksheets #17 nor #18 include a sampling rationale for each sample location. It is not sufficient to include only a general rationale for each sampling area without at least a decision matrix. According to pages 21 and 22 of the UFP QAPP Manual (Volume 2A of the Manual), the text of the SAP should provide a detailed rationale for all sampling locations. Revise Worksheets #17 and #18 to include a detailed rationale for each sampling location. Alternatively, a decision matrix of sufficient detail should be included in the SAP. This decision matrix should allow anyone who has access to the SAP and the information used by the field team to arrive at similar sample locations by following the outlined decision matrix.*

Navy Response to EPA General Comment 3: Worksheet #17 was revised, as follows, to further clarify the sampling rationale:

First paragraph in Section 17.1.1 (Marina AST Area)

> ….. six borings (60SB06 through 60SB11 as shown on Figure 10) will be advanced at this area. Borings 60SB06 through 60SB10 will be advanced at locations approximately 30 feet beyond wells 60SB04 and 60SB05. Due to the presence of the gasoline/diesel fuel ASTs, boring 60SB11 will be advanced at a location approximately 50 feet beyond well 60SB04. If petroleum-impacted soil is identified at an initial location based on PID measurements and/or visual/olfactory observations, an additional boring(s) will be advanced approximately 20 feet progressively farther outward from the impacted boring(s) until the extent of petroleum-impacted soil has been delineated. Conversely, if petroleum-impacted soil is not identified at an initial location based on field observations, an additional boring may be advanced approximately 15 feet farther inward from the initial boring to further refine the extent of impacted soil. The specific distance of the additional radially-spaced borings…..

First paragraph in Section 17.2.2 (Suspected Debris Disposal Areas)

> …..and contingent upon approval from the Navy, USEPA, and PREQB as described above. However, a sufficient number of borings will be advanced at locations adjacent to identified debris within the disposal areas to ensure that worst-case site conditions (i.e., areas posing the greatest potential risk to human health and the environment) will be captured/characterized. In addition, a sufficient number of borings will be advanced at locations outside (i.e., downgradient/ sidegradient/upgradient) of the identified disposal areas to assist in characterization/delineation of potential contamination.

As noted in the same paragraph of Section 17.2.2, the specific number and locations of borings will be based on results of the geophysical survey described in Section 17.2.1 and contingent upon approval from the Navy, USEPA, and PREQB. This approach was agreed to during the project scoping session on September 28 and 29, 2011 (see Worksheet #9).

First paragraph in Section 17.4 (Ensenada Honda)

> …..determine the nature and extent of contamination in the vicinity of these sample locations. Samples 60SD04 through 60SD06 will be located approximately 100 feet radially outward (west, south, and east) from existing sample 60SD01 (Phase I RFI). Samples 60SD07 through 60SD09 will be located approximately 100 feet radially outward (north, west, and south) from existing sample 60SD02 (Phase I RFI).

In addition, Worksheets #18.1 through #18.4 were revised to include a brief description of the sampling location rationale.

EPA General Comment 4: *Step 3 of the project quality objectives (PQOs) states that non-detect constituents will be considered "absent" if they are not detected at the limit of detection (LOD). This approach is not advised because analytes may be present above project action levels (PALs) but are unable to be detected by the analytical method. If reanalysis with appropriately sensitive quantitation limits is not possible, an assessment of the associated uncertainty and impact to the overall estimates of risk and hazard should be provided. Such assessment should address the historic land use and the pragmatic assessment of the potential for the constituent at issue to be present. This will allow EPA to review this data gap assessment and make recommendations for risk management that may include resampling in the face of significant uncertainty.*

Navy Response to EPA General Comment 4: The last paragraph of Section 11.3 in Worksheet #11 (Project Quality Objectives/Systematic Planning Process Statements) was revised, as follows:

> …..detected constituents will be reported to the DL. Non-detect constituents reported at the LOD will be further evaluated qualitatively in the human health and ecological risk assessments, which may be conducted as part of the CMS, or alternatively as part of this RFI if the site characterization is complete. An assessment of the associated uncertainty and impact to the overall estimates of risk and hazard will be provided, as appropriate, to determine if risk management decisions would be affected by the fact the LOD exceeds the PAL for a particular constituent(s).

EPA General Comment 5: *Step 5 of the PQOs states that "(c)onstituent concentrations above the screening values/background values will not automatically designate the SWMU as "dirty" or trigger a response action. However, exceeding a screening value/background value may suggest that further evaluation of the potential risk that may be posed by SWMU-related contaminants is appropriate." It is unclear when further evaluation of constituent concentrations above screening values/background values will be conducted. Revise the SAP to clarify how it will be determined when further evaluation is necessary.*

Navy Response to EPA General Comment 5: For clarity purposes, the following sentence was added to Section 11.3 in Worksheet #11 (Project Quality Objectives/Systematic Planning Process Statements):

> Further evaluation of constituent concentrations above screening values/background values will be conducted in the human health and ecological risk assessments, which may be conducted as part of the CMS, or alternatively as part of this RFI if the site characterization is complete.

EPA General Comment 6: *Step 5 of the PQOs does not provide if…then... statements to address all the questions posed in Step 2. Instead, Step 5 only states that individual constituents will be directly compared to the ecological and human health risk-based screening values and appropriate background values for metals. Revise the PQOs to provide if…then… statements as described in Chapter 5 of EPA's Guidance on Systematic Planning Using the Data Quality Objectives Process (EPA QA/G-4), dated February 2006.*

Navy Response to EPA General Comment 6: Section 11.5 of Worksheet #11 states that "IF a given concentration at a given sampling point exceeds a screening value(s) and the background value (for metals only) for that constituent, THEN that sampling point will be considered to be impacted….." The remaining questions presented in Step 2 (Section 11.2) refer to project-specific needs/considerations necessary to complete the characterization process and are not associated with a type of "action level" or "threshold" that will be used to choose between alternative courses of action. No revisions to the SAP are warranted.

EPA General Comment 7: *The Executive Summary lists the analyte groups that will be targeted for each area under investigation. However, the analyte groups listed in the Executive Summary and Worksheet #11 are not consistent with those listed in Worksheet #18. For example, volatile organic compounds (VOCs), PAHs, pesticides, metals, and TPH in surface and subsurface soil are listed in the Executive Summary for the Marina Aboveground Storage Tank (AST) and disposal areas, but Worksheet #18 does not include analysis of pesticides for the Marina AST area or TPH analysis for the disposal area. Further, pesticides and TPH analyses are included in Worksheet #18 for groundwater, but are not listed in the Executive Summary or Worksheet #11. Revise the SAP to resolve these discrepancies.*

Navy Response to EPA General Comment 7: For clarity purposes, the second bullet of the Executive Summary was revised, as follows:

- Complete the characterization and delineation of: (1) VOCs, PAHs, metals, and TPH in surface and subsurface soil in the vicinity of the former underground piping system associated with the marina's ASTs, (2) VOCs, PAHs, pesticides, and metals in surface and subsurface soil within/near the suspected disposal areas, (3) VOCs, PAHs, and metals in groundwater, and (4) PAHs, pesticides, and metals in sediment within Ensenada Honda adjacent to the SWMU

Similar revisions were made to Sections 11.1 and 11.3 of Worksheet #11. With respect to groundwater, a note was previously included in the table presented in Section 11.3 of the Draft SAP that states "Although pesticides and TPH GRO/DRO were not identified as COPCs in groundwater, they were included in the analytical program due to their presence in soil." No revisions to Worksheet #18 are warranted.

EPA General Comment 8: *Worksheet # 19 indicates that metals in soil, groundwater, and sediment samples will be analyzed by Methods 6020A/6010C. However, the SAP does not specify which analytes will be analyzed by which method, or if all metals will be analyzed by both methods. Further, it is unclear which method will be used to achieve the laboratory-specific limits presented in Worksheet #15. Revise the SAP to clarify this information.*

Navy Response to EPA General Comment 8: Worksheet #19 was revised to indicate that tin in soil, groundwater, and sediment will be analyzed using method 6010C. In addition, Worksheets #15.1 through #15.5 were revised to include the analytical methods.

EPA General Comment 9: *The analytical groups listed for investigation derived waste (IDW) are not consistent between Worksheets #19 and #20. For example, "TCLP VOCs" is listed for solid IDW in Worksheet #19, but is not included in Worksheet #20. Revise the SAP to address this discrepancy.*

Navy Response to EPA General Comment 9: Worksheet #20 was revised to include TCLP VOC, TCLP pesticide, Appendix IX LLPAH, and TPH GRO/DRO analyses for solid IDW and Appendix IX VOC, LLPAH, pesticide, metal, and TPH GRO/DRO analyses for liquid IDW.

SPECIFIC COMMENTS

EPA Specific Comment 1: Worksheet #14, Section 14.2.1, Field Verification of Wetland Boundary Delineation, Page 40: *This section does not provide a comprehensive discussion on whether a full wetland delineation will be performed on jurisdictional and non-jurisdictional wetlands. Also, the section does not discuss minimizing potential wetland impacts during the field sampling activities. Substantive compliance with federal laws and executive orders protecting wetlands require that impacts on wetlands be minimized to the extent feasible during remedy implementation. For additional information on minimizing impacts, see (USEPA, 1994) (Considering Wetlands at CERCLA Sites. Office of Solid Waste and Emergency Response. Publication 9280.0-03;15 EPA540/R-94/019; PB-94-963242). A wetland delineation is recommended at SWMU 60 as well as an impact assessment to determine potential wetland impacts, if any.*

Navy Response to EPA Specific Comment 1: The third sentence of the first paragraph in Section 14.2.1 was revised to indicate that all wetland resource boundaries within the borders of the SWMU will be field-delineated. In addition, the following text was added to the last paragraph in Section 14.2.1:

> During implementation of the field sampling activities, potential wetland impacts will be minimized to the extent feasible. This may include, but not be limited to, clearing only the minimum area necessary for completion of the work and/or using hand tools for sample collection in lieu of heavy equipment.

Wetland impacts during remedy implementation will be evaluated as part of the corrective measures implementation, if warranted.

EPA Specific Comment 2: Worksheet #14, Section 14.2.2, Geophysical Survey, Pages 41-42: *The procedures for the geophysical survey do not include mapping ferrous and metallic surface debris. The global positioning system (GPS) coordinates of ferrous and metallic surface debris should be recorded or logged so that anomalies related to this debris can be discounted when the electromagnetic and magnetic data is interpreted. Revise the SAP to include recording the GPS coordinates of metallic and ferrous surface debris so that this debris can be mapped and discounted when the geophysical survey data is interpreted.*

Navy Response to EPA Specific Comment 2: The geophysical survey procedures detailed in Section 14.2.2 of Worksheet #14 were revised, as follows, to include recording the GPS coordinates of ferrous and metallic debris:

8. The GPS coordinates of ferrous and metallic debris will be recorded/mapped so that anomalies related to this debris can be discounted when the geophysical survey data is interpreted.

EPA Specific Comment 3: Worksheet #14, Section 14.2.4, Monitoring Well Installation and Development, Page 45: *The second paragraph lists the typical limits placed on new MW development; however, there are no clear well development field protocols/stabilization criteria that would be used as indicator parameters for MW development. Revise Worksheet #14 to include field protocols and stabilization criteria for new MW development.*

Navy Response to EPA Specific Comment 3: Section 14.2.4 of Worksheet #14 was revised, as follows, to include well development field protocols/stabilization criteria that will be used for indicator parameters for well development:

> …..to remove fine-grained material and improve the hydraulic efficiency of the well. Water quality parameters (WQPs), including pH, oxidation/reduction potential (ORP), specific conductance, dissolved oxygen, temperature, and turbidity, will be measured throughout the development process. The wells will be developed until the following criteria are met:
>
> - A minimum of three borehole volumes (plus the amount of any water added during the installation process) have been removed.
> - The water is visually clear of sediments [e.g., turbidity is less than 10 Nephelometric Turbidity Units (NTUs)]

- Three successive WQP readings have stabilized within 0.2 Standard Units (S.U.) for pH, 20 millivolts for ORP, 3% for specific conductance, 10% for dissolved oxygen, and 10% for turbidity (when turbidity is greater than 10 NTUs). Although temperature readings will be recorded, they will not be used for stabilization evaluation. Temperatures measured at the surface are affected to some extent by the difference between ambient air and groundwater temperatures and thus can vary over short periods.

It should be noted that it may not always be possible to achieve turbidity measurements less than 10 NTUs due to the presence of clay-rich soils and fine-grained lithologies at NAPR. In the event turbidity measurements less than 10 NTUs cannot be achieved, turbidity will be evaluated based on three successive readings that have stabilized within 10% as indicated above.

EPA Specific Comment 4: Worksheet #17, Section 17.3.1 Monitoring Well Installation and Development, Page 82: *There is no groundwater data presented in the SAP to demonstrate groundwater flow/groundwater gradient direction, and therefore the rationale for MW placement is not clear. The second sentence of this section states that new MWs will be installed at approximately five of the 27 boring locations, and one of the MWs (60SB13 as shown on Figure 10) will be installed downgradient from the former underground piping system associated with the Marina's ASTs. The SAP lacks historical groundwater data so it is not clear why the location of MW 60SB13 is considered downgradient. Additionally, the location of the five proposed monitoring wells are not shown on any of the figures in the SAP. Revise Worksheet #17 and Figure 10 to include groundwater flow/gradient directions, primary and secondary MW locations, and a clear rationale for MW placement.*

Navy Response to EPA Specific Comment 4: Available hydrogeologic information is presented in Worksheet #10 (Conceptual Site Model) of the SAP. Specifically, the Physical Profile – Hydrogeology section of the conceptual site model states that "Hydrogeologic conditions at the SWMU (e.g., groundwater flow direction, hydraulic gradient, hydraulic conductivity, groundwater flow velocity) were not evaluated in the Phase I RFI Report because of uncertainty associated with the linear alignment of the wells. However, expected groundwater flow is south-southwest towards Ensenada Honda." As indicated in Worksheet #11 (Project Quality Objectives/Systematic Planning Process Statements), determination of the hydrogeologic conditions at the SWMU is identified as one of the goals of this Full RFI and required information inputs to resolve the decision statement. Section 17.3.1 of Worksheet #17 indicates that "the new monitoring wells (except well 60SB13) will be located within and downgradient of the suspected debris disposal areas to characterize worst-case groundwater conditions and delineate the extent of contamination." As agreed to during the project scoping session on September 28 and 29, 2011 (see Worksheet #9), the specific number and locations of these wells will be based on results of the geophysical survey described in Section 17.2.1 and contingent upon approval from the Navy, USEPA, and PREQB." Therefore, the well locations are not shown on Figure 10, but the notes on Figure 10 clearly state that "Approximately four additional monitoring wells will be installed and are not shown. The specific number and locations of these wells will be based on results of the geophysical survey." For clarity purposes, the fourth sentence of the first paragraph of Section 17.3.1 was revised to indicate that well 60SB13 will be located in the presumed downgradient direction of the former underground piping system associated with the marina's ASTs. In addition, the presumed groundwater flow direction (south-southwest towards Ensenada Honda) was added to Figure 10.

EPA Specific Comment 5: Worksheet #24, Analytical Instrument Calibration Table, Page 105: *Instrument blanks such as the continuing calibration blank (CCB) are not listed for metals analyses (i.e., ICP-AES, ICP-MS, and CVAA). Instrument blanks should be analyzed for metals analyses to demonstrate that the instrument is free of analytes of interest. Revise this worksheet to include instrument blanks for metals analyses.*

Navy Response to EPA Specific Comment 5: Worksheet #24 was revised to include Initial Calibration Blanks and Continuing Calibration Blanks for the metals analyses (ICP-AES, ICP-MS, and CVAA) to demonstrate that the instruments are free of analytes of interest.

EPA Specific Comment 6: Worksheet #27, Sample Custody Requirements, Page 111: *Section 27.1 indicates that the sample identification for field duplicates will end with "D". However, it is recommended that field duplicates be submitted to the laboratory blind. Revise this table to utilize a different identification system for field duplicates.*

Navy Response to EPA Specific Comment 6: In order to maintain database and sample nomenclature consistency at NAPR, field duplicate samples will continue to be designated with a "D". No revisions to Worksheet #27 are warranted.

EPA Specific Comment 7: Worksheet #28, Laboratory QC Samples Table, Pages 114-124: *This worksheet references the Department of Defense Quality Systems Manual (DoD QSM) 4.3 for both the method/SOP QC acceptance limits and the measurement performance criteria (MPC). However, the laboratory-specific control limits should be provided in the SAP to ensure they will meet the MPC. Revise Worksheet #28 to include the laboratory-specific control limits.*

Navy Response to EPA Specific Comment 7: As indicated in the SAP, CompuChem is a DoD Environmental Laboratory Accreditation Program (ELAP)-certified laboratory, and the Method Acceptance Limits/Measurement Performance Criteria specified in the DoD QSM are the laboratory-specific control limits. Additional details regarding the Method Acceptance Limits/Measurement Performance Criteria are provided in the referenced analytical SOPs (Appendix D). No revisions to Worksheet #28 are warranted.

EPA Specific Comment 8: Worksheet #28.4, Laboratory QC Samples Table, Page 117: *This table states that for metal analysis, a post digestion spike (PDS) will be run when the serial dilution fails or analyte concentration(s) in all samples are <50x the LOD, and the acceptance criteria for the PDS percent recovery (%R) is 75 to 125%. However, Method 6010C indicates that a PDS should be performed when MS/MSD recoveries are unacceptable, and the acceptance criteria for the PDS should be 80 to 120%. Revise this table to indicate that a PDS will also be analyzed whenever MS/MSDs do not meet acceptance limits, and to identify the %R acceptance limits for the PDS as 80 to 120%.*

Navy Response to EPA Specific Comment 8: Worksheet #28.4 was revised to indicate that a post digestion spike will also be analyzed whenever the MS/MSDs do not meet acceptance limits. In addition, the acceptance limits and measurement performance criteria were revised to indicate the percent recovery should be 80-120%.

EPA Specific Comment 9: Worksheet #31, Planned Project Assessments, Page 127: *This worksheet indicates that data audits are performed by the laboratory's QA manager or designee on at least 10% of the QSM 4.2 DoD data packages. However, it is not specified how these data packages are selected for audits (e.g., randomly). Revise the worksheet to clarify this information. Further, this worksheet states that no field audits are planned. The UFP QAPP Manual states that all data generation and collection operations should include at least one field sampling technical systems audit (TSA) at the start of field sampling activities so that effective corrective action measures can be implemented. Revise the SAP to include a field audit for this project or explain how audit results from a similar/on-going NAPR project will be used to satisfy the need for a TSA.*

Navy Response to EPA Specific Comment 9: The following sentence was added to the last paragraph of Worksheet #31 to clarify how DoD data packages are selected for audit:

> The data packages subject to audit are randomly selected by the Laboratory's QA Manager.

In addition, the second paragraph of Worksheet #31 was revised, as follows, to include one field sampling technical systems audit:

> One field sampling technical systems audit will be conducted at the start of field sampling activities so that effective corrective action measures can be implemented if warranted. The audit will be conducted by Baker's Activity Coordinator/Project Manager or appropriate designee. Throughout the field program, Baker's FOL will also monitor field personnel to ensure that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being conducted in accordance with this SAP. The FOL will be responsible for responding to any audit findings and implementing any required corrective action in a timely manner. Baker's Activity Coordinator/Project Manager, Task Manager, QA Officer, and FOL will be responsible for monitoring the effectiveness of any required corrective action implemented.

Worksheet #32 was also revised to reflect the addition of the field sampling technical systems audit.

EPA Specific Comment 10: Worksheet #32, Assessment Findings and Corrective Action Responses, Page 128: *This section does not indicate that EPA will be notified of any significant correct actions that may occur. Revise Worksheet #32 to indicate that EPA will be notified of all significant corrective actions.*

Navy Response to EPA Specific Comment 10: The following sentence was added to Worksheet #32:

> In addition, Baker's Activity Coordinator/Project Manager will notify the Navy, USEPA, and PREQB of any significant corrective actions implemented.

EPA Specific Comment 11: Worksheet #35, Data Validation (Steps IIa and IIb) Process Table, Page 132: *The table indicates that 10% of the raw laboratory data will be reviewed to confirm calibration and quantitation of reported results. However, it is not specified how this raw data is selected for review (e.g., randomly). Revise the table to clarify this information.*

Navy Response to EPA Specific Comment 11: The following sentence was added to Worksheet #35 to specify how the raw data will be selected for review:

> Raw laboratory data for this review will be randomly selected by the validator.

EPA Specific Comment 12: Worksheet #37, Usability Assessment, Page 138: *The completeness section states that a data gap may result if any sample collection activity does not achieve the completeness goal. However, the calculation and completeness goal presented in this section apply to laboratory completeness; no calculation or goal is provided for completeness of sample collection activities. Revise the SAP to provide a completeness goal for sampling activities and to indicate how completeness for sampling activities will be calculated.*

Navy Response to EPA Specific Comment 12: Sample collection completeness is not intended to be calculated as this generally corresponds to the input used in calculating completeness for the analytical data (i.e., number of measurements expected to be collected). For clarity purposes, the second sentence of the last paragraph in Section 37.1.2 was revised to read as follows:

> Any analyte group that does not achieve the completeness goal may be considered a potential data gap.

EPA Specific Comment 13: Figure 4 - Historical Sample Location Map: *Sampling data is not presented for sample locations shown on Figure 4. Historical data should be presented in the SAP and data should the quality of the data should be discussed in the PQOs. If historical sampling data does not meet the PQOs, the SAP should explain why and indicate how the proposed data to be collected will address existing data gaps.*

Navy Response to EPA Specific Comment 13: Historical data from the 1999 Site Characterization, 2004 ECP Investigation, and 2009 Phase I RFI are presented/discussed in Worksheet #10 (Conceptual Site Model). In addition, historical data from the 2009 Phase I RFI (most recent investigation) are presented on Figures 5 through 9 of the SAP. Data from the two older investigations were not presented on figures due to limitations on their use (e.g., data were not validated, data is outdated). These limitations are clearly identified in Worksheet #13 (Secondary Data Criteria and Limitations Table). Furthermore, existing data gaps and the data required to address those data gaps are clearly identified in Worksheet #11 (Project Quality Objectives/Systematic Planning Process Statements). No revisions to the SAP area warranted.

EPA Specific Comment 14: Figure 10 - Proposed Sample and Monitoring Well Location Map: *There is no rationale for surface and subsurface sampling within areas next to the former pipeline alignment. According to the distance scale on Figure 10, it appears surface/subsurface samples are proposed at considerable distances (40 to 50 feet) from the former pipeline alignment. TPH and PAH contamination was confirmed at the surface and in the subsurface at boring locations 60SB04 and 60SB05, which were located within areas close to the pipeline alignment. Surface and subsurface sampling should be focused within areas closer to the pipeline to ensure no sampling data gaps occur. Ten to 15 foot step out borings are recommended to delineate the vertical and lateral extent of contamination along the former pipeline where warranted. Provide a rationale for sampling 40 to 50 feet from the areas of confirmed contamination along the former pipeline or propose additional sampling in areas closer to the pipeline. If this area is being addressed under the SAP for SWMU 74, for clarity and consistency, this should be indicated in the SAP for SWMU 60 and the applicable information should be referenced.*

Navy Response to EPA Specific Comment 14: Figure 10 was revised to show borings 60SB06 through 60SB10 will be advanced at locations approximately 30 feet beyond wells 60SB04 and 60SB05. Due the presence of the gasoline and diesel fuel ASTs, boring 60SB11 will remain at a location approximately 50 feet beyond well 60SB04. In addition, the first paragraph in Section 17.1.1 of Worksheet #17 was revised, as follows, to further clarify the sampling rationale:

> ….. six borings (60SB06 through 60SB11 as shown on Figure 10) will be advanced at this area. Borings 60SB06 through 60SB10 will be advanced at locations approximately 30 feet beyond wells 60SB04 and 60SB05. Due to the presence of the gasoline/diesel fuel ASTs, boring 60SB11 will be advanced at a location approximately 50 feet beyond well 60SB04. If petroleum-impacted soil is identified at an initial location based on PID measurements and/or visual/olfactory observations, an additional boring(s) will be advanced approximately 20 feet progressively farther outward from the impacted boring(s) until the extent of petroleum-impacted soil has been delineated. Conversely, if petroleum-impacted soil is not identified at an initial location based on field observations, an additional boring may be advanced approximately 15 feet farther inward from the initial boring to further refine the extent of impacted soil. The specific distance of the additional radially-spaced borings…..

This area is not being addressed as part of SWMU 74. Therefore, cross references to applicable information are not warranted.

MINOR COMMENTS

EPA Minor Comment 1: Worksheet #5, Project Organizational Chart, Page 17: *It appears that the definitions of the dotted and dashed lines are switched (i.e., dotted lines appear to represent lines of communication and solid lines appear to represent lines of authority). Revise this worksheet if appropriate.*

Navy Response to EPA Minor Comment 1: The legend in Worksheet #5 was revised to indicate that dashed lines represent lines of communication and solid lines represent lines of authority.

EPA Minor Comment 2: Worksheet #19, Field Sampling Requirements Table, Page 95: *The maximum holding time of 28 days for mercury is not listed for the sediment matrix. Revise this table to include the holding time for mercury in the sediment matrix.*

Navy Response to EPA Minor Comment 2: Worksheet #19 was revised to include a maximum holding time of 28 days for mercury analysis of sediment.

PREQB COMMENTS

GENERAL COMMENTS

PREQB General Comment 1: *Once approval of the SAP attained please notify PREQB of the scheduled field activities in order to coordinate field sampling oversight.*

Navy Response to PREQB General Comment 1: Once approval of the SAP is attained, the Navy will notify the PREQB of the scheduled field activities in order to coordinate field sampling oversight (see the Navy's response to PREQB Worksheet-Specific Comment 5).

PREQB General Comment 2: *Please note that the issue of whether groundwater at SWMU 60, as discussed in Worksheet 10 under Ecological Profile, will be addressed upon resolution of PREQB comments concerning the Groundwater Usability Technical Memorandum. Please note that a discussion of groundwater potability is unrelated to the Ecological Profile. Please move the last sentence of that section to a more appropriate section.*

Navy Response to PREQB General Comment 2: The last sentence under Ecological Profile – Relationship of Contaminant Sources to Habitat and Potential Receptors was moved to a new section (Relationship of Contaminant Sources to Potential Receptors) under Land Use and Exposure Profile. In addition, text was added to this new section concerning determination of groundwater usability as per the Groundwater Usability Assessment Technical Memorandum for NAPR dated May 10, 2012. The new section reads as follows:

> Relationship of Contaminant Sources to Potential Receptors – Human receptors could come into direct contact with potential contaminants in surface and shallow subsurface soil, groundwater, or sediment in Ensenada Honda. Although groundwater is shallow (generally between 1.2 and 3 feet bgs), it is not used as a potable source of water at NAPR. Therefore, potential risk due to ingestion of groundwater is low for human receptors. However, this Full RFI and subsequent corrective action determinations involving groundwater will determine groundwater characteristics relative to potable use suitability (see Section 11.2) and will consider potable use of groundwater in the human health risk evaluation as per the Groundwater Usability Assessment Technical Memorandum for NAPR dated May 10, 2012.

WORKSHEET-SPECIFIC COMMENTS

PREQB Worksheet-Specific Comment 1: *Executive Summary, Page 4: An statement regarding results certification by a PR Licensed Chemist should be included in the paragraph that follows the bullet for Sediment Sampling. Although it is stated at Worksheet #29 that Puerto Rico Chemist Certifications are going to be part of the documents and records for the activities, there is not a clear entry on the SAP that stated the certification of the results by a PR Chemist.*

Navy Response to PREQB Worksheet-Specific Comment 1: Clear entries in the SAP that indicate the data will be certified by a Puerto Rico-certified chemist (Miriam Estrada, Analytical Environmental Services International, Inc.) are already provided in Worksheet #14 (third paragraph of Section 14.5 – Laboratory Analysis) and in Worksheet #7 (Personnel Responsibilities Table). However, the following sentence was added to the subject paragraph to further clarify this fact:

> …..to CompuChem in Cary, North Carolina for laboratory analysis. In addition, the data will be certified by a Puerto Rico-certified chemist. Independent, third-party data validation services…..

PREQB Worksheet-Specific Comment 2: *Worksheet #3: Please correct Gloria Toro and Wilmarie Rivera's contact information to the following: Urbanizacion San Jose Industrial Park, 1375 Ponce de Leon, San Juan, Puerto Rico 00926-2604. Also note that in order to contact their by phone, extension 3586 and 6129 needs to be dialed respectively (this comment also affects Worksheet 6).*

Navy Response to PREQB Worksheet-Specific Comment 2: The contact information shown in Worksheet #3 for Gloria Toro and Wilmarie Rivera was revised as indicated. In addition, Worksheet #6 was revised to include the telephone extension for Wilmarie Rivera.

PREQB Worksheet-Specific Comment 3: *Worksheet #10:*

a. *Page 27: Paragraph 6: As it is a stated goal to determine the direction of ground water flow across SWMU 60, please alter the wording the groundwater section of the marina AST Area write-up to reflect that wells MW-1 and MW-4 are in the presumed up gradient direction of the former piping system.*

Navy Response to PREQB Worksheet-Specific Comment 3a: The subject sentence in the groundwater section of the Marina AST Area (Worksheet #10, Release Profile, Contaminant Distribution) was revised to indicate that wells MW-1 and MW-4 are located in the presumed upgradient direction of the former piping system.

b. *Page 28, Future Land Use: Please add potential commercial development to this section and add commercial/industrial workers to the list of future receptors.*

Navy Response to PREQB Worksheet-Specific Comment 3b: The Land Use and Exposure Profile section of Worksheet #10 was revised as follows:

> Potential Future Land Use – …..under future ownership. The site may also be developed for industrial or other commercial purposes.
>
> Potential Future Human Receptors – Marina workers, potential recreational users, trespassers, and industrial/commercial workers

In addition, the section titled "Potential Future Land Use Related Activities" was revised to include industrial or other commercial use-related activities.

c. *Page 28, Ecological Profile:*
   i. *The ecological profile section of this worksheet needs additional information. Under Ecological Receptors and Species of Special Concern please identify Endangered/ Threatened species that may potentially reside and/or forage within the site (including Ensenada Honda).*

Navy Response to PREQB Worksheet-Specific Comment 3c: Additional text discussing the fauna (including species of concern) that may potentially reside and/or forage within SWMU 60 and adjacent habitats (including Ensenada Honda) was included as Appendix H. In addition, the Ecological Profile – Ecological Receptors and Species of Special Concern section of Worksheet #10 was revised to indicate that the subject information is provided in Appendix H.

PREQB Worksheet-Specific Comment 4: *Worksheet 11: Section 11.6: Please clarify the apparent discrepancy between this section and prior sections where this section states that the sampling plan will focus on hot spots but earlier sections indicate that the investigation will determine the nature and extent of contamination, which would require a different approach than only looking at hotspot areas.*

Navy Response to PREQB Worksheet-Specific Comment 4: The first bullet in Section 11.6 of Worksheet #11 was revised as follows:

- Developing a sampling plan based on existing historical data biasing sample collection to known/perceived "hot spots/areas of concern" (i.e., locations within suspected/identified debris disposal areas) and locations outside those "hot spots/areas of concern" (i.e., downgradient/sidegradient/upgradient of suspected/identified debris disposal areas and the former underground piping system associated with the marina's ASTs). By targeting known "hot spots/areas of concern" based on historical data, the sampling approach will ensure that worst-case site conditions (i.e., areas posing the greatest potential risk to human health and the environment) will be captured. In addition, targeting locations outside those "hot spots/areas of concern" will assist in characterization/delineation of potential contamination.

PREQB Worksheet-Specific Comment 5: *Worksheet #14:*

a. *Page 39, Section 14.1.1: The inclusion of coordination with EPA and PREQB regarding field activities is encourage. A notification at least two weeks in advance of beginning sampling and field activities should be given to the agencies so the personnel could coordinate to perform some field oversight.*

Navy Response to PREQB Worksheet-Specific Comment 5a: The following sentence was added to the first paragraph in Section 14.1.1 of Worksheet #14:

In addition, the Navy will notify the USEPA and PREQB approximately two weeks in advance of the scheduled field activities in order to coordinate any agency field oversight.

b. *Page 39, Section 14.1.2: This section does not include that the results will be certificated by a PR Licensed Chemist, although it is stated at Worksheet #29 that Puerto Rico Chemist Certifications are going to be part of the documents and records for the activities, there is not a clear entry on the SAP that stated the certification of the results by a PR Chemist.*

Navy Response to PREQB Worksheet-Specific Comment 5b: Clear entries in the SAP that indicate the data will be certified by a Puerto Rico-certified chemist (Miriam Estrada, Analytical Environmental Services International, Inc.) are already provided in Worksheet #14 (third paragraph of Section 14.5 – Laboratory Analysis) and in Worksheet #7 (Personnel Responsibilities Table). However, the sixth bullet in Section 14.1.2 was revised, as follows, to further clarify this fact:

- Laboratory analysis (CompuChem) including certification of the data by a Puerto Rico-certified chemist (Miriam Estrada, Analytical Environmental Services International, Inc.)

c. *Page 42, Section 14.2.3: It is not clear why the surface and subsurface soil sampling established that soil cores will be collected from each boring continuously in 4-foot increments from the ground surface to the desire depth. Groundwater is expected to be encountered at shallows depths ranging from 1.2 to 3 ft. Surface soil sampling will be from 0 to 1 foot bgs and subsurface soil samples will be from 1 to 3 feet. No more than one 4-foot increment is expected from each core, the rest will serve to install groundwater monitoring where planned.*

Navy Response to PREQB Worksheet-Specific Comment 5c: The word "continuously" was removed from the second sentence of the first paragraph in Section 14.2.3 of Worksheet #14.

d. *Page 45, Section 14.2.5, Third Bullet: The borehole will be capped with soil or concrete at the surface. Please add information regarding on when the borehole will be capped with soil and under what circumstances it will be capped with concrete at the surface.*

Navy Response to PREQB Worksheet-Specific Comment 5d: The third bullet in Section 14.2.5 of Worksheet #14 was revised as follows:

- The borehole will then be backfilled with bentonite grout and capped with concrete at the surface.

e. *Page 46, Section 14.2.5, Paragraph 1: Please provide an indication in the text as to how far from the abandoned well locations the new wells will be installed. A minimum of a five-foot separation and a maximum of a ten-foot separation from the abandoned wells is preferred.*

Navy Response to PREQB Worksheet-Specific Comment 5e: The second paragraph in Section 14.2.5 of Worksheet #14 was revised to indicate that the replacement wells will be installed adjacent to (within 5 to 10 feet) the initial wells.

f. *Page 46, Section 14.2.6, Paragraph 2: Please consider moving this paragraph which discusses the potential for the re-installation of wells in the area of older wells that may not be found, etc. to section 14.2.5. This is an important point and information regarding the presence and condition of the older wells should be obtained long prior to preparing to sample the groundwater.*

Navy Response to PREQB Worksheet-Specific Comment 5f: The subject paragraph in Section 14.2.6 of Worksheet #14 was moved to the end of Section 14.2.5 as suggested.

g. *Page 46, Section 14.2.6, Paragraph 3: Please indicate that the pump intake will be placed at least two feet from the bottom of each monitoring well during groundwater sampling.*

Navy Response to PREQB Worksheet-Specific Comment 5g: The fourth sentence of the subject paragraph in Section 14.2.6 was revised as follows:

…..the pump intake will be placed within the bottom half of the screened interval at least 2 feet from the bottom of the well.

h. *Page 47, Section 14.2.7, Paragraph 1: As decisions regarding the collection of soil samples and installation of monitoring wells will be reserved until such time that the findings of the geophysical survey have been completed in the suspected debris disposal areas, decisions regarding which wells will be subjected to hydraulic testing should also be deferred until that time. Rather than relegating the hydraulic testing to only new wells, it may be prudent to include other existing wells to gather information over a broader area and decisions involving a component of spatial distribution would be better made once the well network has been expanded.*

Navy Response to PREQB Worksheet-Specific Comment 5h: The first paragraph in Section 14.2.7 of Worksheet #14 was revised, as follows, to indicate that slug tests will be conducted at all of the wells (new and existing) except the temporary wells:

> …..and the water level has returned to equilibrium. In addition, slug tests will be conducted at two of the existing wells (MW-1 and MW-4). Slug tests will not be conducted at the remaining three existing wells (6E-01, 6E-02, and 60SB02) since they were constructed as "temporary wells".

Section 17.3.3 (Hydraulic Conductivity Testing) of Worksheet #17 was also revised to include the same.

i. *Page 48, Section 14.2.9: Sediment samples are proposed to be collected from 0 to 4 inches below ground surface (bgs). Section 14.2.1 indicated sediment samples will be collected 0 to 6 inches bgs. The sediment samples should be collected from 0 to 6 inches bgs. If samples are proposed to be collected from 0 to 4 inches bgs, the rationale for this sampling depth should be clearly presented along with sufficient justification. This comment also applies to Section 17.4.*

Navy Response to PREQB Worksheet-Specific Comment 5i: Section 14.2.9 of Worksheet #14 and Section 17.4 of Worksheet #17 were revised to indicate that the sediment samples will be collected from 0 to 0.5 feet bgs. In addition, Worksheet #18.4 was revised to incorporate the 0 to 0.5-foot depth interval for sediment samples.

PREQB Worksheet-Specific Comment 6: *Worksheet 15:*

a. *Worksheets 15.1 and 15.3: Please note the comments on the ecological screening values that were presented in Appendix A for low-level PAHs, pesticides and metals for soil and sediment. Please incorporate these values into these worksheets and re-evaluate PALs and laboratory LOQs and LODs.*

Navy Response to PREQB Worksheet-Specific Comment 6a: PREQB's Appendix A comments regarding both the ecological and human health screening values were noted, and Worksheets #15.1, #15.2, and #15.3 were revised accordingly.

b. *Worksheet #15.2: The listed LODs and LOQs for the PAHs in groundwater using SW-846 method 8270D with selective ion monitoring appear high. Laboratories can typically report down to 0.02 ug/L. Please consider securing a laboratory capable of achieving lower LODs and LOQs for the PAHs. This will ensure the achievement of almost all project action levels for these contaminants of concern. Please note that PREQB is aware of two DoD-approved labs can go down to 0.01 or 0.02 ug/L for PAHs: Alpha Analytical in Westborough, MA or Columbia Analytical Services in Kelso, WA.*

Navy Response to PREQB Worksheet-Specific Comment 6b: CompuChem has already been procured through a competitive bid process to provide laboratory analytical services associated with this SAP. Bids were evaluated based on ability to achieve the PALs/quantitation goals shown on Worksheet #15. Furthermore, the analytical methods shown on Worksheet #19 have been selected in an effort to attain, to the greatest extent practicable, LOQs, LODs, and DLs less than or equal to the most stringent PALs shown on Worksheet #15. However, based on the limitations of best available technology, CompuChem's quantitation limits (as well as quantitation limits for most if not all other laboratories) for some constituents still do not meet the associated PALs as shown on Worksheet #15. As stated in Section 11.3 of Worksheet #11, analytical results for detected constituents that have a LOQ, LOD, and DL greater than the PAL will be reported to the DL. Non-detect constituents reported at the LOD will be further evaluated qualitatively in the human health and ecological risk assessments, which may be conducted as part of the CMS, or alternatively as part of this RFI if the site characterization is complete. An assessment of the associated uncertainty and impact to the overall estimates of risk and hazard will be provided, as appropriate, to determine if risk management decisions would be affected by the fact the LOD exceeds the PAL for a particular constituent(s).

Columbia Analytical Services has been included in the bid process for recent work associated with other SWMUs at NAPR, and their quantitation limits are comparable to CompuChem's.

c. *Please clarify which RSL is listed in this table (residential or industrial) as both are listed in Appendix A. Note also it would be helpful to indicate the value represents the adjusted RSL.*

Navy Response to PREQB Worksheet-Specific Comment 6c: For clarity purposes, the following sentence was added to the notes of Worksheets #15.1 and #15.3:

> Human health RSLs represent values for residential soil; noncarcinogenic RSLs based on a target hazard quotient of 0.1 for conservative screening purposes (see Appendix A).

In addition, the following sentence was added to the notes of Worksheet #15.2:

> Human health RSLs represent values for tapwater; noncarcinogenic RSLs based on a target hazard quotient of 0.1 for conservative screening purposes (see Appendix A).

PREQB Worksheet-Specific Comment 7: *Worksheet #17, Section 17.1.1, First Paragraph: It is established that six soil borings (60SB06 through 60SB11) will be advanced at locations approximately 40 to 50 feet beyond wells 60SB04 and 60SB05. Meanwhile, four additional soil borings (60SB12 through 60SB15) are anticipated to be required, if necessary to complete the delineation. The ten soil borings are presented in Figure 10 with the same symbol. It is recommended to use a different symbol for the borings that will be actually advanced and for the borings that may be required at the mentioned figure.*

Navy Response to PREQB Worksheet-Specific Comment 7: Figure 10 was revised as indicated to differentiate between borings that actually will be advanced and contingency borings.

PREQB Worksheet-Specific Comment 8: *Worksheet #17,*

a. *Section 17.1.1, Second Paragraph: Please add "at Section 14.2.3" to the last sentence. In order, to clearly state that the specific sampling collection and field screening procedures are detailed on Section 14.2.3 of Worksheet #14.*

Navy Response to PREQB Worksheet-Specific Comment 8a: The subject sentence in the second paragraph of Section 17.1.1 was revised as follows:

> Specific sample collection and field screening procedures are detailed on Worksheet #14 (Section 14.2.3).

b. *Suspected Debris Disposal Areas: Please clarify what actions will be taken to identify the nature of anomalies identified during the geophysical investigation.*

Navy Response to PREQB Worksheet-Specific Comment 8b: The fourth paragraph in Section 17.2.1 of Worksheet #17 was revised, as follows, to clarify what actions will be taken to identify the nature of anomalies identified during the geophysical investigation:

> …..using conventional survey methods. In addition, any surficial anomalies will be visibly inspected to identify the nature of the anomalies (e.g., type of anomaly/debris; condition; presence of liquids, sludges, solids in containers).

Section 14.2.2 (Geophysical Survey) of Worksheet #14 was also revised to include the same.

c. *Please clarify why trenching is not proposed subsequent to the geophysical investigation. It seems trenching is warranted to evaluate the nature of the debris to ensure that sources of contamination will not remain in place resulting in future releases and to support adequate characterization with the reduced analyte list, especially considering the typically heterogeneous nature of disposal areas.*

Navy Response to PREQB Worksheet-Specific Comment 8c: Currently, it is unknown whether buried debris exists at the site. However, it is important to note that the presence of buried debris is unlikely given the shallow depth to groundwater (1.2 to 3 feet bgs). The Navy concurs that trenching may be warranted to evaluate the nature of any buried debris (if present) and support adequate characterization of the site. If buried debris is identified as a result of the geophysical survey, trenching activities may be proposed as a subsequent phase of investigation.

PREQB Worksheet-Specific Comment 9: *Worksheet 18.3: Please revise the sample location numbers for wells 60SB-4R and 60SB05R to include the "R".*

Navy Response to PREQB Worksheet-Specific Comment 9: Worksheet #18.3 was revised to include the "R" designation for wells 60SB04 and 60SB05 since they will be replaced.

PREQB Worksheet-Specific Comment 10: *Worksheet 19:*

a. *Please clarify why SW-846 method 9040C is referenced for the pH analysis of soil samples instead of SW-846 method 9045D which is specifically used for the pH analysis of soil samples.*

Navy Response to PREQB Worksheet-Specific Comment 10a: Worksheets #19 and #28.11 were revised to include pH analysis of soil samples using Method 9045D. In addition, the associated laboratory SOP (SOP 3.5.14.3) was included in Appendix D.

b. *For the groundwater analyses of PAHs, pesticides and DRO, please revise the number of containers collected to two-1 liter amber glass bottles for each analysis instead of one bottle for each analysis. This allows for potential re-extractions due to surrogate issues, bottle breakage, etc.*

Navy Response to PREQB Worksheet-Specific Comment 10b: Worksheet #19 was revised to include two 1-liter glass amber containers for PAH, pesticide, and TPH DRO analyses of groundwater.

c. *For the TOC analysis of sediment, please change the method reference to Lloyd Kahn instead of 9060A to be consistent with EPA Region 2 requirements.*

d. *For the TOC analysis of sediment, please change the holding time to 14 days to be consistent with Lloyd Kahn requirements.*

Navy Response to PREQB Worksheet-Specific Comment 10c and 10d: Worksheet #19 was revised to include the Lloyd Kahn method and a maximum holding time of 14 days for TOC analysis of sediment. In addition, Worksheet #28.8 was revised to include the Lloyd Kahn method.

e. *For the metals analysis of sediment, please include the holding time for mercury.*

Navy Response to PREQB Worksheet-Specific Comment 10e: Worksheet #19 was revised to include a maximum holding time of 28 days for mercury analysis of sediment.

f. *For the containers, volume and preservation requirements for solid IDW for PAHs, GRO and DRO, please revise the information to reflect solid samples instead of aqueous samples.*

Navy Response to PREQB Worksheet-Specific Comment 10f: The containers, sample volumes, preservation requirements, and maximum holding times for PAH and TPH GRO/DRO analyses of solid IDW was revised to reflect a solid matrix in lieu of an aqueous matrix.

g. *For the liquid IDW analyses of PAHs, pesticides and DRO, the number of containers collected should be revised to two-1 liter amber glass bottles for each analysis instead of one bottle for each analysis. This allows for potential re-extractions due to surrogate issues, bottle breakage, etc.*

Navy Response to PREQB Worksheet-Specific Comment 10g: Worksheet #19 was revised to include two 1-liter glass amber containers for PAH, pesticide, and TPH DRO analyses of liquid IDW.

PREQB Worksheet-Specific Comment 11: *Worksheet 20: Please add the missing analyses for solid IDW and liquid IDW to this worksheet.*

Navy Response to PREQB Worksheet-Specific Comment 11: Worksheet #20 was revised to include TCLP VOC, TCLP pesticide, Appendix IX LLPAH, and TPH GRO/DRO analyses for solid IDW and Appendix IX VOC, LLPAH, pesticide, metal, and TPH GRO/DRO analyses for liquid IDW.

PREQB Worksheet-Specific Comment 12: *Worksheet 21: Please include the SOPs that will be utilized for all sampling procedures, including but not limited to well installation, well development, soil sampling, equipment decontamination, sediment sampling, headspace measurements, water quality parameter measurements, etc. Although some of the procedures were described in the QAPP, detailed SOPs are required to ensure standardized sampling procedures between samplers, the collection of representative samples, and to ensure data comparability and usability. As per the UFP QAPP Manual, sampling procedures should include SOPs for sampling each matrix and each analytical parameter for each type of equipment and technique. Please include all required SOPs in Appendix C of the revised report for agency review.*

Navy Response to PREQB Worksheet-Specific Comment 12: Detailed sampling and data collection procedures specific to this SAP are presented in Worksheet #14. As per the UFP QAPP Manual, sampling procedures that will be used in the project must be documented in the SAP OR attached documents (i.e., SOPs). A comprehensive description of the sampling and data collection procedures is an acceptable form of documentation. The Navy believes that the sampling and data collection procedures presented in Worksheet #14 are detailed to a level that will provide consistency between samplers; facilitate collection of accurate, precise, and representative samples; and help to ensure data comparability and usability. No revisions to the SAP are warranted.

PREQB Worksheet-Specific Comment 13: *Worksheet 23: Please provide the method numbers and SOPs that will be utilized for the preparation of soil, sediment and groundwater samples for PAH, pesticide, DRO and metals analyses. Please include all SOPs in Appendix D.*

Navy Response to PREQB Worksheet-Specific Comment 13: Worksheet #23 was revised to include the method numbers and SOPs that will be utilized for the preparation of soil, sediment, and groundwater samples for LLPAH, pesticide, TPH DRO, and metals analyses. Worksheet #19 was also revised to include the same. In addition, the subject SOPs were included in Appendix D.

PREQB Worksheet-Specific Comment 14: *Worksheet 24: The acceptance criteria for the continuing calibration verification associated with GRO analysis is listed as "percent difference/drift must be ≤20%." However, the SOP requires that the percent difference/drift be ≤15%. Please revise accordingly.*

Navy Response to PREQB Worksheet-Specific Comment 14: The acceptance criteria shown on Worksheet #24 for the continuing calibration verification associated with TPH GRO analysis was revised to indicate that the percent difference/drift must be ≤ 15%.

PREQB Worksheet-Specific Comment 15: *Worksheet 27:*

a. *Please revise Section 27.2 to eliminate the use of clear tape over labels for VOC and GRO bottles for soil and groundwater samples. The use of clear tape can affect the final weights of soil samples and can also interfere with the robotics of autosamplers for both soil and groundwater samples.*

Navy Response to PREQB Worksheet-Specific Comment 15a: The first paragraph in Section 27.2 of Worksheet #27 was revised, as follows, to eliminate the use of clear tape over labels on VOC and TPH GRO containers:

> In addition, clear tape will be placed over the labels (except on VOC and TPH GRO containers) to preserve the information. Clear tape will not be placed over the labels on VOC and TPH GRO containers for soil and groundwater samples because the tape can affect the final weights of soil samples and interfere with the robotics of auto-samplers for both soil and groundwater samples.

b. *In Section 27.4, please clarify that the pH of aqueous samples for VOC or GRO analysis will not be checked by the Sample Custodian upon receipt at the laboratory. The pH of these samples is typically checked by the analyst after the analysis has been performed so as to not compromise the samples.*

Navy Response to PREQB Worksheet-Specific Comment 15b: The last paragraph in Section 27.4 of Worksheet #27 was revised as follows:

> …..and/or infrared thermometer. In addition, the Sample Custodian will check the pH values for aqueous samples (except samples for VOC and TPH GRO analyses) requiring preservative to ensure that they are within the acceptable range. The pH of aqueous samples for VOC and TPH GRO analyses will be checked by the analyst after the analysis has been conducted so as to not compromise the samples. Any discrepancies…..

PREQB Worksheet-Specific Comment 16: Worksheet 28:

a. *Worksheet 28.1: Please add the RPD acceptance criteria for the MSD.*

Navy Response to PREQB Worksheet-Specific Comment 16a: Worksheet #28.1 was revised to include a RPD acceptance criteria of ≤ 30% for the MSD.

b. *Worksheet 28.4: Please eliminate the reference to common laboratory contaminants.*

Navy Response to PREQB Worksheet-Specific Comment 16b: Worksheet #28.4 was revised to eliminate reference to common laboratory contaminants for the method blank.

c. *Worksheet 28.4: Please revise the internal standard acceptance criteria to be in accordance with the DoD requirements listed in the laboratory SOP: 30-120% of the internal standard in*

Navy Response to PREQB Worksheet-Specific Comment 16c: The internal standard acceptance criteria listed on Worksheet #28.4 was revised to indicate that the absolute response must not deviate more than 30-120% of the original response in accordance with DoD requirements listed in the laboratory SOP.

APPENDIX A – SCREENING VALUES

PREQB Appendix A Comment 1: *Please provide a reference for the residential and industrial RSLs, including the relevant date, and indicate whether they are adjusted RSLs (i.e., noncarcinogenic values reduced by a factor of 10 to account for cumulative effects at the screening stage). Please ensure that the most current version of the RSL table is referenced (November 2011).*

Navy Response to PREQB Appendix A Comment 1: A reference for the USEPA RSLs was added to the screening value tables provided in Appendix A. The most current RSLs (May 2012) were used, and Worksheets #15.1, #15.2, and #15.3 were updated accordingly (see the Navy's response to PREQB Worksheet-Specific Comment 6a). In addition, the following note was added to the screening value tables provided in Appendix A to indicate that the RSLs were reduced by a factor of 10:

> Noncarcinogenic Regional Screening Levels based on a target hazard quotient of 0.1 for conservative screening purposes.

PREQB Appendix A Comment 2: *Please clarify the rationale for selecting the ecological screening values presented in these tables. The hierarchy of selecting soil, groundwater and sediment ecological screening values needs to be presented for each medium. For example, in selecting screening values for the soil medium, the first reference source used should based on screening values presented in the USEPA eco-SSL documents followed by the appropriate secondary sources (e.g., USEPA Region 5 soil ecological screening levels) if an eco-SSL is unavailable. The next reference sources should be provided in order of their preference. Please add this hierarchy as footnotes for the soil, groundwater and sediment tables in Appendix A. The identification of the hierarchy for selection of screening values ensures that a systematic and transparent method was applied in the selection of these values.*

Navy Response to PREQB Appendix A Comment 2: Narrative text was included in Appendix A to clarify the rationale/hierarchy for selecting the ecological screening values for soil, groundwater, and sediment.

PREQB Appendix A Comment 3: *Ecological screening values (eco-SSLs) are available for PAHs detected in soil and need to be added for these contaminants. Typically, the sum of low molecular weight (LMW) and high molecular weight (HMW) PAHs are used as the ecological screening benchmark*

Navy Response to PREQB Appendix A Comment 3: Appendix A was revised to include the ecological screening values for Low Molecular Weight PAHs (29,000 µg/kg) and High Molecular Weight PAHs (18,000 µg/kg) for soil.

PREQB Appendix A Comment 4: *Ecological screening values for groundwater based on the USEPA ECOTOX database should present the study endpoint and the appropriate uncertainty factors used to select the screening value. For example, if an LC-50 result was selected from the database, the applicable uncertainty factor to derive a chronic NOAEL needs to be presented.*

Navy Response to PREQB Appendix A Comment 4: Appendix A was revised to include a table that presents the study endpoint and the appropriate uncertainty factors used to select the ecological screening values for groundwater.

FINAL SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
FULL RCRA FACILITY INVESTIGATION SWMU 60 – FORMER LANDFILL AT THE MARINA

For NAVAL ACTIVITY PUERTO RICO
EPA I.D. No. PR2170027203
CEIBA, PUERTO RICO

Prepared for:

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Contract No. N62470-10-D-3000
DO JM07

June 7, 2012

IDIQ for A/E Services for Multi-Media Environmental Compliance Engineering Support

SAP Worksheet #1: Title and Approval Page

FINAL

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
FULL RCRA FACILITY INVESTIGATION
SWMU 60 – FORMER LANDFILL AT THE MARINA

NAVAL ACTIVITY PUERTO RICO
EPA I.D. NO: PR2170027203
CEIBA, PUERTO RICO

June 7, 2012

Prepared for:
DEPARTMENT OF THE NAVY
NAVFAC SOUTHEAST
North Charleston, South Carolina

Prepared by:
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Prepared under:
CONTRACT NO. N62470-10-D-3000
DELIVERY ORDER JM07

Review Signature: _____ 6/7/12
Richard Aschenbrenner Date
Baker, Program QA Officer

Approval Signature: See attached for Navy Chemist Signature
Jon Tucker Date
NAVFAC Atlantic, Chemist/QA Officer

Other Approval Signature: _____ 6/7/11
Mark Davidson Date
Navy BRAC PMO SE/BEC

SAP Worksheet #1: Title and Approval Page

DRAFT

SAMPLING AND ANALYSIS PLAN
(Field Sampling Plan and Quality Assurance Project Plan)
FULL RCRA FACILITY INVESTIGATION
SWMU 60 – FORMER LANDFILL AT THE MARINA

NAVAL ACTIVITY PUERTO RICO
EPA I.D. NO: PR2170027203
CEIBA, PUERTO RICO

December 30, 2011

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North Charleston, South Carolina

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Prepared under:
CONTRACT NO. N62470-10-D-3000
DELIVERY ORDER JM07

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I certify under penalty of law that I have examined and am familiar with the information submitted in this document and all attachments and that this document and its attachments were prepared either by me personally or under my direction or supervision in a manner designed to ensure that qualified and knowledgeable personnel properly gather and present the information contained therein. I further certify, based on my personal knowledge or on my inquiry of those individuals immediately responsible for obtaining the information, that the information is true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fines and imprisonment for knowingly and willfully submitting a materially false statement.

Signature: _____

Name: **Mark E. Davidson**

Title: **BRAC Env. Coordinator**

Date: **June 7, 2012**

EXECUTIVE SUMMARY

Michael Baker Jr., Inc. (Baker) has been retained by the U.S. Department of the Navy, Base Realignment and Closure (BRAC) Program Management Office (PMO) Southeast (SE) under contract with the Naval Facilities Engineering Command (NAVFAC), (Contract Number N62470-10-D-3000, Delivery Order [DO] JM07) to conduct a Full Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at Solid Waste Management Unit (SWMU) 60 – Former Landfill at the Marina at Naval Activity Puerto Rico (NAPR), Ceiba, Puerto Rico.

SWMU 60 is located adjacent to Ensenada Honda near the southeastern portion of NAPR. The SWMU was historically used as a debris disposal area from the 1940s to 1960s. In addition, a portion of the SWMU was used as a small marina beginning sometime between 1958 and 1961. Currently, a large marina (constructed in the 1970s; upgraded in the mid1990s) covers the majority of the SWMU and consists of the marina building, concrete boat slips and docks, two aboveground storage tanks (ASTs), concrete walkways, manicured lawn, tennis courts, and asphalt roads and parking. The marina ceased operations as of September 2011. The southeastern portion of the SWMU is covered with secondary growth vegetation and is described as an estuarine intertidal scrub-shrub broad-leaved evergreen (E2SS3) wetland. A small, elevated beach area classified as marine intertidal unconsolidated shore sand (M2US2) is located on the southern edge.

On January 29, 2007, the U.S. Environmental Protection Agency (USEPA) issued a RCRA §7003 Administrative Order on Consent (USEPA, 2007), which identified documented releases of solid and/or hazardous waste and hazardous constituents at SWMU 60 (formerly referred to as Environmental Condition of Property [ECP] Site 6). The Administrative Order required completion of an equivalent Phase I RFI, which was conducted in January 2009 (Baker, 2011). Based on results of this investigation, it was determined that a Full RFI was warranted due to the presence of volatile organic compounds (VOCs), polynuclear aromatic hydrocarbons (PAHs), pesticides, metals, and/or total petroleum hydrocarbons (TPH) in soil, groundwater, and sediment at the SWMU.

This document presents the Sampling and Analysis Plan (SAP) (Field Sampling Plan and Quality Assurance Project Plan) for the Full RFI proposed to be conducted at SWMU 60. The SAP was prepared in accordance with the requirements of the *Uniform Federal Policy (UFP) for Quality Assurance Project Plans* (USEPA, 2005) and USEPA *Guidance for Quality Assurance Project Plans* (USEPA, 2002). The 37 worksheets identified in Part 2A of the UFP guidance are presented herein. In addition, figures and appendices that support the information presented on the worksheets are included.

The primary goal of the Full RFI is to collect additional data required to complete the site characterization process and support the decision for Corrective Action Complete (CAC) or further action. Specifically, data collected during the Full RFI will be of the quantity and quality necessary to achieve the following objectives:

- Determine the boundary limits of the suspected debris disposal areas and depth of debris (if buried)

- Complete the characterization and delineation of: (1) VOCs, PAHs, metals, and TPH in surface and subsurface soil in the vicinity of the former underground piping system associated with the marina's ASTs, (2) VOCs, PAHs, pesticides, and metals in surface and subsurface soil within/near the suspected disposal areas, (3) VOCs, PAHs, and metals in groundwater, and (4) PAHs, pesticides, and metals in sediment within Ensenada Honda adjacent to the SWMU

- Determine the hydrogeologic conditions at the SWMU (e.g., groundwater flow direction, hydraulic gradient, hydraulic conductivity, groundwater flow velocity)

- Determine the interaction between shallow groundwater and Ensenada Honda

For investigation purposes, the SWMU has been divided into the following four areas based on history and site layout, potential source areas, release mechanisms, and contaminant distribution: (1) marina AST area, (2) suspected debris disposal areas, (3) SWMU-wide groundwater, and (4) Ensenada Honda. Sampling and data collection activities proposed to be conducted at each of these areas will primarily include the following:

Marina AST Area

- Surface and subsurface soil sampling
- Monitoring well abandonment and replacement

Suspected Debris Disposal Areas

- Field verification of wetland boundary delineation
- Geophysical survey
- Surface and subsurface soil sampling

SWMU-Wide Groundwater

- Monitoring well installation and development
- Groundwater sampling
- Hydraulic conductivity testing
- Evaluation of the interaction between shallow groundwater and Ensenada Honda

Ensenada Honda

- Sediment sampling

The sampling and data collection activities will be conducted by qualified field personnel in accordance with procedures specified in this SAP. The samples, including appropriate field quality assurance/quality control (QA/QC) samples, will be submitted to CompuChem in Cary, North Carolina for laboratory analysis. In addition, the data will be certified by a Puerto Rico-certified chemist. Independent, third-party data validation services will be provided by DataQual Environmental Services, LLC (DataQual) of St. Louis, Missouri. The laboratory analytical data (except solid and liquid investigation derived waste data) will be evaluated to assess the technical adequacy and usability of the data. Upon completion of validation and evaluation of the data, a Full RFI report will be prepared. In general, the report will include, but not be limited to, the following:

- Introduction including regulatory framework and objectives of the Full RFI
- Background information
- Description of the physical characteristics of the study area
- Description of the sampling and data collection activities
- Discussion of current site conditions
- Presentation and interpretation of the SWMU-specific geologic and hydrogeologic conditions
- Presentation and interpretation of the analytical results

- Summary of the data validation/usability assessment
- Discussion of the site conceptual model
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ACRONYMS AND ABBREVIATIONS

A/E	Architectural and Engineering
AES	Atomic Emission Spectroscopy
AMU	Atomic Mass Units
AOCs	Areas of Concern
APA	Aerial Photographic Analysis
ASTs	Aboveground Storage Tanks
ASTM	American Society for Testing and Materials
Baker	Michael Baker Jr., Inc.
BEC	BRAC Environmental Coordinator
bgs	below ground surface
BRAC	Base Realignment and Closure
BTEX	Benzene, Toluene, Ethylbenzene, and Xylenes
C	Celsius
CAC	Corrective Action Complete
CADD	Computer Aided Design and Drafting
CA LUFT	California Leaking Underground Fuel Tank
CCB	Continuing Calibration Blank
CCCs	Calibration Check Compounds
CCME	Canadian Council of Ministers of the Environment
CCV	Continuing Calibration Verification
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
CMS	Corrective Measures Study
COA	Certificate of Analysis
COPCs	Constituents of Potential Concern
CSM	Conceptual Site Model
CVAA	Cold Vapor Atomic Adsorption
DataQual	DataQual Environmental Services, LLC
DGPS	Differential Global Positioning System
DLs	Detection Limits
DoD	Department of Defense
DI	Deionized
DPT	Direct Push Technology
DQI	Data Quality Indicator
DQOs	Data Quality Objectives
DRO	Diesel Range Organics
ECD	Electron Capture Detection
ECP	Environmental Condition or Property
EDD	Electronic Data Deliverable
ELAP	Environmental Laboratory Accreditation Program
EM	Electromagnetic
ERA	Ecological Risk Assessment
F	Fahrenheit
FID	Flame Ionization Detector
FOL	Field Operations Leader

ACRONYMS AND ABBREVIATIONS
(continued)

GC/MS	Gas Chromatograph/Mass Spectrometer
GIS	Geographic Information System
GPS	Global Positioning System
GRO	Gasoline Range Organics
HASP	Health and Safety Plan
HDOP	Horizontal Dilution of Precision
HHRA	Human Health Risk Assessment
ICAL	Initial Calibration
ICB	Initial Calibration Blank
ICP	Inductively Coupled Plasma
ICS	Interference Check Solution
ID	Inside Diameter
IDW	Investigation Derived Waste
IQC	Indefinite Quantity Contract
LCS	Laboratory Control Sample
LIMS	Laboratory Information Management System
LLPAHs	Low-Level Polynuclear Aromatic Hydrocarbons
LNAPL	Light, Non-Aqueous Phase Liquid
LODs	Limits of Detection
LOQs	Limits of Quantitation
MCAWW	Methods for Chemical Analysis of Water and Wastes
MCL	Maximum Contaminant Level
MHSPE	Ministry of Housing, Spatial Planning, and Environment
mg/kg	milligrams per kilogram
ml/min	milliliters per minute
MS	Matrix Spike
MSD	Matrix Spike Duplicate
msl	mean sea level
NA	Not Applicable
NAD	North American Datum
NAPR	Naval Activity Puerto Rico
NAVFAC	Naval Facilities Engineering Command
NAVY	U.S. Department of the Navy
NDIRD	Non-Dispersive Infrared Detector
NTUs	Nephelometric Turbidity Units
ORP	Oxidation/Reduction Potential
%C	Percent Complete
%R	Percent Recovery
PAHs	Polynuclear Aromatic Hydrocarbons
PALs	Project Action Limits

ACRONYMS AND ABBREVIATIONS
(continued)

PCBs	Polychlorinated Biphenyls
PDF	Portable Document Format
PDOP	Position Dilution of Precision
PI	Photo Identified
PID	Photoionization
PMO	Project Management Office
POCs	Points of Contact
PQOs	Project Quality Objectives
ppm	parts per million
PREQB	Puerto Rico Environmental Quality Board
PRWQS	Puerto Rico Water Quality Regulations
PT	Proficiency Testing (previously known as performance evaluation [PE] sample)
PVC	Polyvinyl Chloride
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
QSM	Quality Systems Manual
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RFI	RCRA Facility Investigation
RPD	Relative Percent Difference
RPM	Remedial Project Manager
RSD	Relative Standard Deviation
RSL	Regional Screening Level
RWEC	Right Way Environmental Contractors, Inc.
SAP	Sampling and Analysis Plan
SDG	Sample Delivery Group
SE	Southeast
SHSO	Site Health and Safety Officer
SIM	Selected Ion Monitoring
SNR	Signal to Noise Ratio
SOPs	Standard Operating Procedures
SPCCs	System Performance Check Compounds
S.U.	Standard Units
SVOA	Semivolatile Organic Analytes
SVOCs	Semivolatile Organic Compounds
SWMU	Solid Waste Management Unit
TBD	To Be Determined
TCLP	Toxicity Characteristic Leaching Procedure
TDS	Total Dissolved Solids
TOC	Total Organic Carbon
TPH	Total Petroleum Hydrocarbons
TSS	Total Suspended Solids

ACRONYMS AND ABBREVIATIONS
(continued)

UFP	Uniform Federal Policy
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
ULM	Upper Limit of the Means
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
VOA	Volatile Organic Analytes
VOCs	Volatile Organic Compounds
WAAS	Wide Area Augmentation System
WQPs	Water Quality Parameters

SAP Worksheet #2: Sampling and Analysis Plan Identifying Information

Site Name/Number: Naval Activity Puerto Rico (NAPR), Solid Waste Management Unit (SWMU) 60 – Former Landfill at the Marina

Operable Unit: Not Applicable (NA)

Contractor Name: Michael Baker Jr., Inc. (Baker)

Contract Number: N62470-10-D-3000

Contract Title: Indefinite Quantity Contract (IQC) for Architectural and Engineering (A/E) Services for Multi-Media Environmental Compliance Engineering Support

Delivery Order Number: JM07

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP)* (U.S. Environmental Protection Agency [USEPA], 2005) and USEPA *Guidance for Quality Assurance Project Plans* (USEPA, 2002).

2. Identify regulatory program: Resource Conservation and Recovery Act (RCRA)

3. This SAP is a project-specific SAP.

4. List organizational partners (stakeholders) and identify the connection with lead organization:

Organization Partners/Stakeholders	Connection
USEPA Region II	Lead regulatory agency overseeing RCRA activities implemented at NAPR by lead organization
Puerto Rico Environmental Quality Board (PREQB)	Regulatory stakeholder overseeing RCRA activities implemented at NAPR by lead organization
Naval Facilities Engineering Command (NAVFAC)	Implements environmental restoration activities at NAPR

5. Lead organization: NAVFAC

6. If any required SAP elements and required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below:

   - The crosswalk table has been excluded since all required information is provided in this SAP.

SAP Worksheet #3: Distribution List

SAP Recipients	Title	Organization	Telephone Number	E-mail Address and/or Mailing Address
Adolph Everett	Chief, RCRA Programs Branch	USEPA Region II	212-637-4109	290 Broadway, 22nd Floor New York, NY 10007-1866
Timothy Gordon	Project Coordinator	USEPA Region II RCRA Programs Branch	212-637-4167	290 Broadway, 22nd Floor New York, NY 10007-1866 gordon.timothy@epamail.epa.gov
Carl Soderberg	Director, Caribbean Environmental Protection Division	USEPA, Caribbean Environmental Protection Division	787-729-6951	Centro Europa Building, Suite 417 1492 Ponce de Leon Avenue Santurce, PR 00907-4127
Brenda Smith	USEPA Contractor	TechLaw, Inc.	703-818-1000	14500 Avion Parkway, Suite 300 Chantilly, VA 20151
Wilmarie Rivera-Otero	Federal Facilities Coordinator	PREQB	787-767-8181 x 6129	Urbanizacion San Jose Industrial Park 1375 Ponce de Leon San Juan, PR 00926-2604 wilmarierivera@jca.gobierno.pr
Gloria Toro-Agrait	Environmental Permits Officer	PREQB Hazardous Wastes Permit Division	787-767-8181 x 3586	Urbanizacion San Jose Industrial Park 1375 Ponce de Leon San Juan, PR 00926-2604 gloriatoro@jca.gobierno.pr
Felix Lopez	Liaison	U.S. Fish & Wildlife Service	787-851-7297	Road 301 km 5.1 Boqueron, PR 00622
Mark Davidson	Base Realignment and Closure (BRAC) Environmental Coordinator (BEC)	U.S. Department of the Navy (Navy) BRAC Project Management Office (PMO) Southeast (SE)	843-743-2124	4130 Faber Place Drive, Suite 202 North Charleston, SC 29405 mark.e.davidson@navy.mil
Stacin Martin	Remedial Project Manager (RPM)	NAVFAC Atlantic	757-322-4780	6506 Hampton Boulevard Norfolk, VA 23508-1278 stacin.martin@navy.mil
Bonnie Capito	Librarian and Records Manager	NAVFAC Atlantic	757-322-4785	6506 Hampton Boulevard Norfolk, VA 23508-1278
Pedro Ruiz	NAPR Environmental Manager	NAVFAC Atlantic	757-286-9139	NAPR, Building 2439 Ceiba, PR 00735 pedro.ruiz1@navy.mil
Mark Kimes (shared copy with Baker project team personnel)	Activity Coordinator/Project Manager	Baker	412-269-2009	Airside Business Park, 100 Airside Drive Moon Township, PA 15108 mkimes@mbakercorp.com

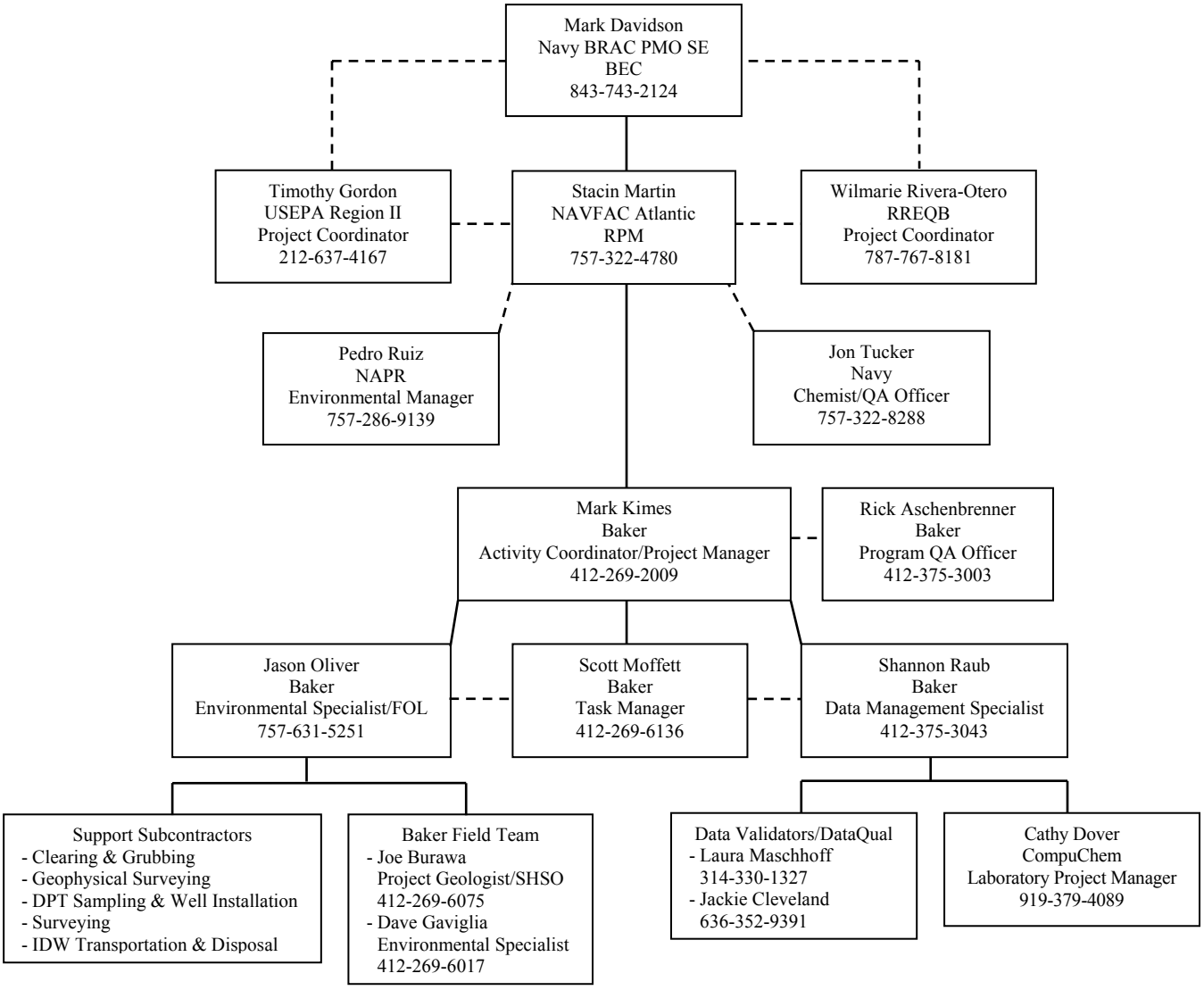
SAP Worksheet #3: Distribution List (*continued*)

SAP Recipients	Title	Organization	Telephone Number	E-mail Address and/or Mailing Address
Cathy Dover	Laboratory Project Manager	CompuChem	919-379-4089	501 Madison Avenue Cary, NC 27513 cdover@compuchemlabs.com
Laura Maschhoff	Data Validator - Organics	DataQual Environmental Services, LLC (DataQual)	314-330-1327	5830 Amberway Drive St. Louis, MO 63128 dataqual@charter.net
Jackie Cleveland	Data Validator - Inorganics	DataQual	636-352-9391	137 Royallprarie Lane O'Fallon, MO 63368 cleve137@charter.net

SAP Worksheet #4: Project Personnel Sign-Off Sheet

Name	Title/Role	Organization	Telephone Number (optional)	Signature/E-mail Receipt	SAP Section Reviewed	Date SAP Reviewed
Mark Kimes	Activity Coordinator/Project Manager	Baker	412-269-2009	Mark E. Kimes	All	11/23/2011
Rick Aschenbrenner	Program Quality Assurance (QA) Officer	Baker	412-375-3003	See Worksheet #1	All	11/15/2011
Scott Moffett	Geologist/Task Manager	Baker	412-269-6136		All	11/16/2011
Jason Oliver	Environmental Specialist/Field Operations Leader (FOL)	Baker	757-631-5251			
Joe Burawa	Project Geologist/Site Health and Safety Officer (SHSO)	Baker	412-269-6075			
Dave Gaviglia	Environmental Specialist/Field Team Member	Baker	412-269-6017			
Shannon Raub	Data Management Specialist/Data Tracking and Management	Baker	412-375-3043			
Cathy Dover	Laboratory Project Manager	CompuChem	919-379-4089			
Laura Maschhoff	Data Validator – Organics	DataQual	314-330-1327			
Jackie Cleveland	Data Validator - Inorganics	DataQual	636-352-9391			

SAP Worksheet #5: Project Organizational Chart



SAP Worksheet #6: Communication Pathways

Communication Drivers	Responsible Entity	Name	Telephone Number and/or E-mail Address	Procedure (timing, pathway to/from, etc.)
Navy Interface (e.g., submission of SAP for review, receipt of regulatory comments, etc.)	BEC RPM	Mark Davidson Stacin Martin	843-743-2124 mark.e.davidson@navy.mil 757-322-4780 stacin.martin@navy.mil	Communication to/from the project team via e-mail, telephone, hardcopy, or in-person as warranted; can delegate communication to other internal or external Point of Contacts (POCs); RPM to report any significant corrective actions to the regulatory agencies involved.
SAP Changes prior to Field/ Laboratory Work				Any major changes to this SAP must be approved by the Navy, USEPA, and PREQB Project Coordinators prior to implementation. Changes will be communicated/discussed via e-mail and/or telephone. Upon agreement, the SAP will be revised and distributed as shown on Worksheets #3 and #4.
Regulatory Agency Interface – USEPA (e.g., receipt of SAP for review; submission of USEPA comments)	USEPA Project Coordinator	Timothy Gordon	212-637-4167 gordon.timothy@epamail.epa.gov	Communication to/from the project team via e-mail, telephone, hardcopy, or in-person as warranted; can delegate communication to other internal or external POCs.
Regulatory Agency Interface – PREQB (e.g., receipt of SAP for review; submission of PREQB comments)	PREQB Project Coordinator	Wilmarie Rivera-Otero	787-767-8181 x 6129 wilmarierivera@jca.gobierno.pr	Communication to/from the project team via e-mail, telephone, hardcopy, or in-person as warranted; can delegate communication to other internal or external POCs.
Project Administration and Logistics (e.g., submission of documents for review, receipt of Navy/regulator comments, updates on project progress, communication of stakeholder expectations, etc.)	Baker Activity Coordinator/Project Manager	Mark Kimes	412-269-2009 mkimes@mbakercorp.com	Primary liaison to BRAC PMO SE RPM (Mark Davidson) via e-mail, telephone, hardcopy, or in-person as warranted; direct communication to/from project staff to ensure appropriate project implementation; may delegate communication to other staff as appropriate.
Project Logistics (e.g., coordination of field activities, updates on project progress, etc.)	Baker Task Manager	Scott Moffett	412-269-6136 smoffett@mbakercorp.com	Direct communication via e-mail, telephone, hardcopy, or in-person, as warranted, to/from project staff to ensure appropriate project implementation and adherence to this SAP.
Daily Field Progress Reports	Baker FOL	Jason Oliver	757-631-5251 joliver@mbakercorp.com	The FOL will e-mail daily field progress reports to the Baker Project Manager, QA Officer, Task Manager, and Data Management Specialist upon completion of work each day. Direct communication via telephone and/or e-mail will be conducted on an as-needed basis.
SAP Changes in the Field				Deviations from this SAP will be discussed with (via telephone) and subject to approval from the Baker Project Manager, QA Officer, and Task Manager before they are executed. Such deviations will be communicated to the project team as soon as practicable upon realization that a change may be warranted but before any change is executed. Any major changes to this SAP must be approved by the Navy, USEPA, and PREQB Project Coordinators prior to implementation as previously noted. Deviations and appropriate rationale will be recorded in the field logbook(s) and included in the Daily Field Progress Reports.
Field Corrective Actions				Any field corrective actions will be immediately addressed in the field (see Worksheet #32)

SAP Worksheet #6: Communication Pathways (*continued*)

Communication Drivers	Responsible Entity	Name	Telephone Number and/or E-mail Address	Procedure (timing, pathway to/from, etc.)
Work Stoppage Issues	Baker FOL Baker Activity Coordinator/Project Manager	Jason Oliver Mark Kimes	757-631-5251 joliver@mbakercorp.com 412-269-2009 mkimes@mbakercorp.com	If field circumstances warrant stoppage of work for reasons including, but not limited to, health and safety or weather-related concerns, the Baker FOL will verbally inform the Project Manager and Task Manager within one hour. Work will not resume until the issue has been resolved. The Baker Project Manager will inform (verbally or via email) the Navy BEC and RPM of any work stoppage within one day.
Health and Safety Expectations and Procedures	Baker SHSO	Joe Burawa	412-269-6075 jburawa@mbakercorp.com	Review of site-specific Health and Safety Plan (HASP) prior to initiation of the field activities; direct communication via e-mail, telephone, hardcopy, or in-person, as warranted, to/from Baker project team (including subcontractors) to ensure implementation of appropriate health and safety procedures.
Ensuring Staff Health and Safety in the Field				Daily safety tailgate meetings; daily observations; real-time discussions of observations and changes to be implemented with field staff.
Consultation with the Navy, USEPA, and PREQB Regarding Placement of some Borings/Monitoring Wells	Baker Activity Coordinator/Project Manager Baker Task Manager	Mark Kimes Scott Moffett	412-269-2009 mkimes@mbakercorp.com 412-269-6136 smoffett@mbakercorp.com	Upon evaluation of the geophysical survey results, the specific number and locations of proposed borings/monitoring wells within and near the suspected debris disposal areas will be discussed and agreed upon with the Navy, USEPA, and PREQB. It is anticipated that this will be accomplished via an on-site meeting and/or teleconference during the field program (see Worksheets #14 and #17).
Data Tracking/Sample Receipt Variances	Baker Data Management Specialist	Shannon Raub	412-375-3043 shannon.raub@mbakercorp.com	The Baker Data Management Specialist will track data from sample collection through upload to the project database to ensure requirements of this SAP are met by the field team and laboratory. Tracking will involve receipt of daily sample summaries from the FOL, receipt of daily sample log-in reports from the Laboratory Project Manager, and receipt of electronic and hardcopy data from the laboratory and data validator. The Baker Data Management Specialist will communicate via e-mail, telephone, or in-person, as warranted, with the Project Manager, QA Officer, Task Manager, FOL, Laboratory Project Manager, and data validator to ensure adherence to project analysis and validation requirements.
Reporting Laboratory Quality Variances	Laboratory Project Manager	Cathy Dover	919-379-4089 cdover@compuchemlabs.com	Laboratory issues pertaining to the field samples will be reported by the Laboratory Project Manager to the Baker Project Manager, QA Officer, Task Manager, and Data Management Specialist via e-mail within two business days. Direct communication via telephone and/or e-mail will be conducted on an as-needed basis.
Analytical Corrective Actions				Analytical corrective actions (See Worksheets #24, #25, #28, and #32) will be reported via telephone and/or e-mail within two business days from the time the issue is identified.
Reporting Data Validation Issues/ Corrective Actions	DataQual	Laura Maschhoff Jackie Cleveland	314-330-1327 dataqual@charter.net 636-352-9391 cleve137@charter.net	Data validation issues/corrective actions will be reported by the validator to the Baker Project Manager, QA Officer, Task Manager, and Data Management Specialist via telephone and/or e-mail within two business days from the time the issue is identified. Direct communication via telephone and/or e-mail will be conducted on an as-needed basis.

SAP Worksheet #7: Personnel Responsibilities Table

Organizational Affiliation	Name	Title/Role	Responsibilities
Navy BRAC PMO SE	Mark Davidson	BEC	Coordination/oversight of BRAC environmental restoration activities implemented at NAPR
NAVFAC Atlantic	Stacin Martin	RPM	Coordination/oversight of BRAC environmental restoration activities implemented under this SAP
	Jon Tucker	Chemist/QA Officer	Navy review of SAP and QA input
	Pedro Ruiz	NAPR Environmental Manager	On-island Navy liaison; provide logistical support for BRAC environmental restoration activities implemented under this SAP
Baker	Mark Kimes	Activity Coordinator/Project Manager	Program support to ensure consistency with NAPR expectations/requirements; project administration and oversight; monitor project performance; coordinate staffing; direct and oversee project staff
	Rick Aschenbrenner	Program QA Officer	Oversee adherence to program and project-specific QA and quality control (QC) requirements; document review
	Scott Moffett	Geologist/Task Manager	Technical lead; direct and oversee project staff; coordinate field activities and supervise field sampling; ensure adherence to this SAP; data evaluation and report development
	Shannon Raub	Data Management Specialist/Data Tracking and Management	Primary communications with laboratory and data validator; manage sample tracking; assimilation of data from field collection through analysis, validation, and upload to the project database; perform queries for data evaluation and report development
	Jason Oliver	Environmental Specialist/FOL	Ensure on-site compliance with this SAP; coordinate field activities and supervise field sampling; assist with field sampling; sample management/shipping in the field; daily field progress reports
	Joe Burawa	Project Geologist/SHSO	Implement field activities in accordance with this SAP; oversee and ensure safety of on-site personnel
	Dave Gaviglia	Environmental Specialist/Field Team Member	Implement field activities in accordance with this SAP; field verification of wetland boundary delineation
CompuChem	Cathy Dover	Laboratory Project Manager	Laboratory POC; overall management of project-specific analytical work under this SAP
	James Feldhaus	Laboratory QA Officer	Laboratory QA program and review of QC data
	Bradley Lake	Organics Laboratory Department Manager	Oversight of organics laboratory; QC and analytical data review
	Joe Mohn	Inorganics Laboratory Department Manager	Oversight of inorganics laboratory; QC and analytical data review
Analytical Environmental Services International, Inc.	Miriam Estrada	Puerto Rican Chemist	Certification of laboratory analytical data
DataQual	Laura Maschhoff	Data Validator - Organics	Validation of organic analytical data
	Jackie Cleveland	Data Validator - Inorganics	Validation of inorganic analytical data

SAP Worksheet #7: Personnel Responsibilities Table (*continued*)

Organizational Affiliation	Name	Title/Role	Responsibilities
Right Way Environmental Contractors, Inc. (RWEC)	Pedro Tejada	Clearing and Grubbing Subcontractor	Clear and grub vegetation, as necessary, to access geophysical survey and sample locations
To Be Determined (TBD)	TBD	Geophysical Surveying Subcontractor	Conduct geophysical survey; data evaluation/interpretation
TBD	TBD	Drilling Subcontractor	Direct push technology (DPT) sampling and monitoring well installation
TBD	TBD	Surveying Subcontractor	Horizontal coordinate and vertical elevation survey of boring and monitoring well locations
TBD	TBD	Investigation Derived Waste (IDW) Transportation and Disposal Subcontractor	Off-site transportation and disposal of IDW

Notes:
Resumes are maintained by the individuals' organizations and are available upon request. Upon execution of the project, staff may be added, as necessary, or substituted based on availability.
Geophysical surveying, drilling, surveying, and IDW transportation and disposal subcontractors are subject to availability at the time the field program is implemented and will be procured prior to implementation.

SAP Worksheet #8: Special Personnel Training Requirements Table

Project Function	Specialized Training by Title or Description of Course	Training Provider	Training Date	Personnel/Groups Receiving Training	Personnel Titles/ Organizational Affiliation	Location of Training Records/Certificates
No specialized/non-routine, project-specific training requirements or certifications are needed by personnel in order to successfully complete this project. The sampling will be conducted by experienced geologists. Analytical services will be provided by a Department of Defense (DoD) Environmental Laboratory Accreditation Program (ELAP)-certified and Navy-approved laboratory (CompuChem). Other subcontracted services will be provided by qualified subcontractors.						

SAP Worksheet #9: Project Scoping Session Participants Sheet

Project Name: Full RCRA Facility Investigation (RFI)

Site Name: NAPR; SWMU 60 – Former Landfill at the Marina

Projected Date(s) of Sampling: Summer 2012

Site Location: Ceiba, Puerto Rico

Project Manager: Mark Kimes

Date of Session: September 28 and 29, 2011

Scoping Session Purpose: Site visit to SWMU 60 to familiarize the project team with the site layout; meeting to discuss the scope and technical approach for conducting the Full RFI

Name	Title	Affiliation	Telephone Number and/or E-mail Address	Project Role
Timothy Gordon	Project Coordinator	USEPA Region II RCRA Programs Branch	212-637-4167 gordon.timothy@epamail.epa.gov	USEPA regulator and lead POC; oversight of RCRA activities implemented at NAPR
Cathy Dare (via telephone; 2nd day only)	USEPA Contractor	TechLaw, Inc.	703-818-1000	Document technical review on behalf of USEPA
Travis Kline (via telephone; 2nd day only)	USEPA Contractor	TechLaw, Inc.	703-818-1000	Document technical review on behalf of USEPA
Tom Paul (via telephone; 2nd day only)	USEPA Contractor	TechLaw, Inc.	703-818-1000	Document technical review on behalf of USEPA
Wilmarie Rivera-Otero	Federal Facilities Coordinator	PREQB	787-767-8181 wilmarierivera@jca.gobierno.pr	PREQB regulator and lead POC; oversight of RCRA activities implemented at NAPR
Gloria Toro-Agrait	Environmental Permits Officer	PREQB Hazardous Wastes Permit Division	787-767-8181 gloriatoro@jca.gobierno.pr	PREQB regulator – document technical review
Mark Davidson	BEC	Navy BRAC PMO SE	843-743-2124 mark.e.davidson@navy.mil	Coordination/oversight of BRAC environmental restoration activities implemented at NAPR
Stacin Martin	RPM	NAVFAC Atlantic	757-322-4780 stacin.martin@navy.mill	BRAC PMO SE support; coordination/oversight of BRAC environmental restoration activities implemented under this SAP
Pedro Ruiz	NAPR Environmental Manager	NAVFAC Atlantic	757-286-9139 pedro.ruiz1@navy.mil	On-island Navy liaison
Mark Kimes	Activity Coordinator/Project Manager	Baker	412-269-2009 mkimes@mbakercorp.com	Program support to ensure consistency with NAPR expectations/requirements; project administration and oversight
Scott Moffett	Geologist/Task Manager	Baker	412-269-6136 smoffett@mbakercorp.com	Technical lead; preparation of SAP

SAP Worksheet #9: Project Scoping Session Participants Sheet (*continued*)

Comments/Decisions:

- The well screens for existing monitoring wells near the former underground piping system associated with the marina's above ground storage tanks (ASTs) (60SB04 and 60SB05) were installed below the water table. Therefore, the wells are not suitable to assess the presence/absence of light, non-aqueous phase liquid (LNAPL). Consider replacing these two wells or installing piezometers that intersect the water table.

- The specific number of boring and well locations within/near the suspected debris disposal areas will be based on results of the geophysical survey. Therefore, upon completion of the geophysical survey, consider preparing a memo and recommending specific boring/well locations as a step in the SAP. This may be discussed via teleconference with the Navy, USEPA, and PREQB while in the field to minimize costs associated with subsequent mobilizations.

- Consider collecting groundwater elevations from existing wells at adjacent SWMUs, in addition to wells at SWMU 60, to provide a more comprehensive understanding of the groundwater flow regime.

- Need to ensure sufficient data is collected to support the ecological and human health risk assessment process. The risk assessments may be conducted as part of the Full RFI in lieu of the Corrective Measures Study (CMS) if the site characterization is complete.

Action Items:

- Prepare UFP-SAP in accordance with agreed upon scope and technical approach.

Consensus Decisions:

- Semivolatile organic compounds (SVOCs) can be eliminated from the analytical program since they were not detected during previous investigations; include only low-level polynuclear aromatic hydrocarbons (LLPAHs).

- Include VOCs in the analytical program for all of the soils.

- Need to further investigate polynuclear aromatic hydrocarbons (PAHs), pesticides, and metals in sediment near samples 6E-SD01, 60SD01, and 60SD02 (Ensenada Honda) even though these impacts may not be attributed to a release from the SWMU.

SAP Worksheet #10: Conceptual Site Model

Profile Type	Information Needed	Findings
Site Profile	Installation Name	NAPR
	Installation Location	Ceiba, Puerto Rico
	Site Name	SWMU 60 – Former Landfill at the Marina
	Site Location	SWMU 60 is located adjacent to Ensenada Honda just southwest of the former athletic fields and Tow Way Drive (see Figures 1 through 3).
	Site History	SWMU 60 was historically used as a debris disposal area from the 1940s to 1960s. A portion of the site was used as a small marina beginning sometime between 1958 and 1961. Currently, a large marina (constructed in the 1970s; upgraded in the mid 1990s) covers the majority of the site. The marina ceased operations as of September 2011.
	Previous Investigations	Previous investigation activities are summarized below; the secondary data criteria and limitations are provided on Worksheet #13. The sample and monitoring well locations are shown on Figure 4; a summary of results is presented in the Release Profile, Contaminant Distribution section of this CSM. **1999 Site Characterization** (CH2M Hill, 1999) - Focused on investigating potential petroleum-related impacts associated with the marina's former underground piping system due to a reported release that occurred prior to 1999 (exact date unknown) - Piping system was removed sometime after 1999 (exact date unknown) - 12 soil borings (SB-1 through SB-12) – samples analyzed for benzene, toluene, ethylbenzene, and xylenes (BTEX) and total petroleum hydrocarbons (TPH) gasoline range organics (GRO)/diesel range organics (DRO) - Four new wells (MW-1 through MW-4) – samples analyzed for BTEX, TPH GRO/DRO, PAHs, and lead **2004 Phase I Environmental Condition of Property (ECP) Investigation** (NAVFAC Atlantic, 2005) - Aerial Photography Analysis (APA) – photo identified (PI) Site 9 (ECP Site 6) due to observation of solid waste and scrap metal disposal area - Disturbed ground or ground scarring and probable debris or refuse were only identified in 1958 aerial photograph (three polygon features as shown on Figure 3) - Records review and interviews confirmed the site was historically used as a debris disposal area - It is unknown whether buried debris exists at the site; however, presence of buried debris is unlikely given the shallow depth to groundwater (1.2 to 3 feet below ground surface [bgs]) - Physical site inspection: only small quantities of scrap metal were observed in the area; the majority of the site is now covered by the marina; no evidence of staining or stressed vegetation **2004 Phase II ECP Investigation** (NAVFAC Atlantic, 2005) - Three soil borings (6E-01 through 6E-03) - Two new wells (6E-01 and 6E-02) - Two surface water/sediment samples from Ensenada Honda (6E-SW/SD01 and 6E-SW/SD02) - Samples analyzed for Appendix IX volatile organic compounds (VOCs), SVOCs, pesticides, polychlorinated biphenyls (PCBs), organophosphorous pesticides, chlorinated herbicides, and metals

SAP Worksheet #10: Conceptual Site Model (*continued*)

Site Profile (*continued*)	Previous Investigations (*continued*)	**2009 Phase I RFI** (Baker, 2011) - Focused on determining whether or not there has been a release from the SWMU - Five soil borings (60SB01 through 60SB05) ▪ Five surface soil samples (0-1 foot bgs) ▪ Subsurface soil samples: three from 1-3 feet bgs and one from 5-7 feet bgs ▪ Samples analyzed for Appendix IX VOCs, SVOCs/LLPAHs, pesticides, metals, and/or TPH GRO/DRO - Three sediment samples from Ensenada Honda (60SD01 through 60SD03) – samples analyzed for Appendix IX SVOCs/LLPAHs, pesticides, and metals - Three groundwater samples from one new temporary well (60SB02) and two new permanent wells (60SB04 and 60SB05) – samples analyzed for Appendix IX VOCs, SVOCs/LLPAHs, pesticides, metals, and TPH GRO/DRO
	Site Area and Layout	SWMU 60 consists of an approximate 13-acre area and includes two distinct portions as shown on Figure 3 and described below: 1. The marina covers the majority of the site (approximately 10.5 acres) and consists of the marina building, concrete boat slips and docks along a large portion of the shoreline, two ASTs, concrete walkways, manicured lawn, tennis courts, and asphalt roads and parking. 2. The southeastern portion (approximately 2.5 acres) consists of a level area and extends approximately 60 feet into Ensenada Honda. The land area is covered with secondary growth vegetation including red mangrove. This area is described as estuarine intertidal scrub-shrub broad-leaved evergreen (E2SS3) wetland. Tidal influence within this E2SS3 wetland unit has not been evaluated. A small, elevated beach area classified as marine intertidal unconsolidated shore sand (M2US2) is located on the southern edge. The paved entrance road located on the eastern side of the marina is a generalized boundary between the developed marina area and the secondary growth undeveloped area. Evidence of surface debris, including tires, wood, and metal, was observed in this undeveloped area during the Phase I ECP Investigation and Phase I RFI. No evidence of metal or other debris was observed in the developed marina area.
	Site Structures	A large portion of the SWMU 60 shoreline has been developed into boat slips and docks associated with the marina. The marina also consists of the marina building, two ASTs, concrete walkways, manicured lawn, tennis courts, and asphalt roads and parking.
	Potential Sources	Historical records indicate that, prior to 1999 (exact date unknown), there was a release of petroleum products from the former underground piping system associated with the marina's ASTs, which are located in the west-central portion of the SWMU (see Figure 3). Debris associated with the three suspected disposal areas in the eastern, central, and southwestern portions of the SWMU also constitutes a source.
	Release Mechanisms	Historical release of petroleum products to soil. Releases of solid and/or hazardous waste and hazardous constituents associated with debris to soil. Potential contaminant migration to groundwater/surface water.
	Site Security	SWMU 60 is located within NAPR, which currently is a secure Navy facility. Currently, restricted access via guard at Gate 3 is the only entry point to NAPR. NAPR security personnel monitor all access to NAPR, and frequent motor vehicle and boat patrols are conducted as part of the access control program. There is no SWMU-specific security.
Physical Profile	Climate	Puerto Rico has a tropical marine climate characterized by minimal temperature fluctuations, relatively moderate humidity, and frequent rain showers. Mean annual maximum temperatures range from 82.0° Fahrenheit (F) in January to 88.2° F in August. The mean annual minimum temperatures vary from 64.0° F in January to 73.2° F in June. The rainy season is typically defined as May through November with an average of 60 inches of rain per year although a dry winter season occurs from December through April. Hurricane season is from June 1 through November 30.
	Topography	The topography is generally flat with ground elevations ranging from near mean sea level (msl) to approximately 9 feet above msl (100 to 109 feet datum; note that the datum plan used is the Mean Low Water plus 100.00 feet as established by the U.S. Navy Survey Section [November 1941]).
	Geology	The unconsolidated materials near the western portion of the SWMU (60SB04 and 60SB05) consist primarily of sand with varying amounts of silt, clay, or gravel. Peat and sandy clay are more predominant near the eastern portion of the SWMU (60SB02 and 60SB03). During the Phase I RFI, it was difficult to distinguish between fill material and natural deposits, but the area may consist of all fill material that was brought in to construct the marina area. Weathered bedrock (4.3 feet bgs) was only encountered in boring 60SB01.

SAP Worksheet #10: Conceptual Site Model (*continued*)

Physical Profile (*continued*)	Hydrogeology	Groundwater was observed during the Phase I RFI at shallow depths generally between 1.2 and 3 feet bgs. The proximity of SWMU 60 to the shoreline of Ensenada Honda accounts for the shallow groundwater occurrence. Hydrogeologic conditions at the SWMU (e.g., groundwater flow direction, hydraulic gradient, hydraulic conductivity, groundwater flow velocity) were not evaluated in the Phase I RFI Report because of uncertainty associated with the linear alignment of the wells. However, expected groundwater flow is south-southwest towards Ensenada Honda. The concrete slips and docks may be causing some mounding of shallow groundwater near the shoreline where these structures exist. However, the interaction between shallow groundwater and Ensenada Honda has not been evaluated.
	Vegetation	The majority of the SWMU is occupied by the marina, and little vegetation exists other than manicured lawn areas and a few trees. The southeastern, undeveloped portion of the SWMU is covered with secondary growth vegetation including red mangrove. This area is predominantly described as estuarine intertidal scrub-shrub broad-leaved evergreen (E2SS3).
Release Profile	Contaminant Distribution	The distribution of contaminants at the SWMU is discussed below. Constituents detected at concentrations exceeding the screening values during the 2009 Phase I RFI (most recent investigation) are shown on Figures 5 through 9. **Marina AST Area** Soil – Based on results from the 1999 Site Characterization, petroleum-related impacts (BTEX/TPH GRO) were identified in surface and shallow subsurface soil (SB-3 through SB-6 as shown on Figure 4) in the vicinity of the former underground piping system associated with the marina's ASTs. The soil contamination appeared to be limited to the area between the ASTs and Ensenada Honda. Subsequent samples collected from this area in 2009 (Phase I RFI) showed elevated PAHs (60SB04) and arsenic (60SB05) were present in surface soil (see Figure 5). Elevated benzo(a)pyrene was also present in shallow subsurface soil (60SB04) (see Figure 6). In addition, TPH DRO and/or GRO (60SB04 and 60SB05) were present in subsurface soil at concentrations exceeding the PREQB screening value and extended to 7 feet bgs (below the water table) in boring 60SB04. Groundwater – Elevated benzene was present in groundwater from well MW-3 (1999 Site Characterization). In addition, elevated PAHs and lead were present in wells MW-1 through MW-4. Wells MW-1 and MW-4 are located in the presumed upgradient direction of the former piping system, which suggests that the presence of these constituents may not be entirely related to the former piping system. Subsequent samples collected from this area in 2009 (Phase I RFI) showed elevated arsenic (60SB04) and PAHs (60SB05) were present in groundwater (see Figure 9). LNAPL was not identified in the wells. However, wells closest to the former piping system (60SB04 and 60SB05) are screened below the water table and not suitable to assess the presence/absence of LNAPL. **Suspected Debris Disposal Areas** Surface Soil – Elevated benzo(a)pyrene (60SB02) and arsenic (60SB01 and 60SB02) were present in surface soil from the suspected disposal area near the eastern portion of the SWMU (see Figure 5). In addition, elevated benzo(a)pyrene and a number of metals (60SB03) were present in surface soil from the suspected disposal area near the central portion of the SWMU. Subsurface Soil – Arsenic (6E-SB02 and 6E-SB03) was the only constituent present at elevated concentrations in shallow subsurface soil from the suspected disposal areas near the eastern and southwestern portions of the SWMU. Three elevated PAHs, 4,4'-DDE, and a number of metals (60SB03) were present in shallow subsurface soil from the suspected disposal area near the central portion of the SWMU (see Figures 6 and 7). Groundwater – Based on the limited data available, elevated barium (6E-02 and 60SB02) and vanadium (6E-01) were present in groundwater.

SAP Worksheet #10: Conceptual Site Model (*continued*)

Release Profile (*continued*)	Contaminant Distribution (*continued*)	**Ensenada Honda** Surface Water – No Constituents of Potential Concern (COPCs) Sediment – A number of PAHs, 4,4'-DDE, arsenic, copper, mercury, and tin exceeded the marine sediment screening values in sample 6E-SD01 (Phase II ECP Investigation). This sample was located among several docks that have been used for boat parking/refueling. Furthermore, a sail boat was noted to have burned near this location while it was docked a few months before the samples were collected. Tin was the only constituent that exceeded screening values in sample 6E-SD02. Subsequent samples collected in 2009 (Phase I RFI) showed elevated PAHs and arsenic were present in sediment south of the marina (60SD01) (see Figure 8). Elevated benzo(a)pyrene, dibenz(a,h)anthracene, and arsenic were also present in sample 60SD02, which was located west of the marina and sample 6E-SD01. Although elevated PAHs, 4,4'-DDE, and metals were present in sediment, their occurrence likely is attributed to anthropogenic sources and not a release from the SWMU based on the following: - The site has been used as a marina since at least 1961 - A large portion of the shoreline is used for boat slips/docks/refueling - A sail boat was noted to have burned while it was docked - 4,4'-DDE likely is an artifact of historical pesticide application - Surface run-off from the developed marina area is conveyed to Outfall NR-009, which is located near the southern end of the boat slip/dock area (see Figure 3) - Sheet flow into Ensenada Honda from the undeveloped portion of the SWMU is prevented by the elevated beach area **Conclusion** – The nature and extent of contamination in surface and subsurface soil, groundwater, and sediment at the SWMU has not been delineated, and the boundary limits of the suspected debris disposal areas have not been adequately investigated.
Land Use and Exposure Profile	Current Land Use	Currently, the majority of land area is occupied by the marina (marina building, concrete boat slips and docks, concrete walkways, manicured lawns, tennis courts, and asphalt roads and parking). The marina ceased operations in September 2011. The remaining portion of the SWMU consists of undeveloped, estuarine intertidal scrub-shrub broad-leaved evergreen (E2SS3).
	Current Human Receptors	Potential receptors include Navy personnel, contractors/visitors, and trespassers.
	Current Activities	Currently, no activities take place on the site.
	Potential Future Land Use	SWMU 60 is under investigation, and the need to restrict media and land uses will be determined at the conclusion of the investigation. The site will remain under the ownership and control of the Navy until response actions are complete. However, it is anticipated that the marina will be operated under future ownership. The site may also be developed for industrial or other commercial purposes.
	Potential Future Human Receptors	Marina workers, potential recreational users, trespassers, and industrial/commercial workers
	Potential Future Land Use Related Activities	Commercial/recreational land use associated with the marina; industrial or other commercial use-related activities
	Zoning/Land Use Restrictions	There are no SWMU-specific restrictions at this time pending outcome of the subject Full RFI.
	Demographics/ Zoning	Ceiba's population density is approximately 230 people per square mile. Only a skeleton crew has remained at NAPR since its closure in 2004.
	Beneficial Resources	Other than the marina, Ensenada Honda, and the E2SS3 wetland unit, there are no SWMU 60-specific beneficial resources.
	Relationship of Contaminant Sources to Potential Receptors	Human receptors could come into direct contact with potential contaminants in surface and shallow subsurface soil, groundwater, or sediment in Ensenada Honda. Although groundwater is shallow (generally between 1.2 and 3 feet bgs), it is not used as a potable source of water at NAPR. Therefore, potential risk due to ingestion of groundwater is low for human receptors. However, this Full RFI and subsequent corrective action determinations involving groundwater will determine groundwater characteristics relative to potable use suitability (see Section 11.2) and will consider potable use of groundwater in the human health risk evaluation as per the Groundwater Usability Assessment Technical Memorandum for NAPR dated May 10, 2012.

SAP Worksheet #10: Conceptual Site Model (*continued*)

Ecological Profile	Habitat Type	The majority of SWMU 60 is comprised of the marina (marina building, concrete boat slips and docks, concrete walkways, manicured lawn, tennis courts, and asphalt roads and parking), which is not typically considered habitat. The southeastern, undeveloped portion is covered with secondary growth vegetation including red mangrove. This area is described as estuarine intertidal scrub-shrub broad-leaved evergreen (E2SS3). A small beach classified as marine intertidal unconsolidated shore sand (M2US2) is located on the southern edge of the site.
	Degree of Disturbance	Moderate – Historically, the site was used as a debris disposal area. However, disposal operations ceased in the 1960s, and the habitat and ecological receptors present will remain the same.
	Ecological Receptors and Species of Special Concern	The fauna (including species of concern) that may potentially reside and/or forage within SWMU 60 and adjacent habitats are identified and discussed in Appendix H.
	Relationship of Contaminant Sources to Habitat and Potential Receptors	Ecological receptors could come into direct contact with potential contaminants in surface and shallow subsurface soil, groundwater, or sediment in Ensenada Honda. Ecological receptors could come into contact with potential contaminants while digging for food or constructing burrows, or that have been incorporated into the food chain (bioaccumulated in plants and animals).

SAP Worksheet #11: Project Quality Objectives/Systematic Planning Process Statements

The project quality objectives (PQOs) described below were developed using the USEPA seven-step data quality objectives (DQOs) process presented in *Guidance on Systematic Planning using the Data Quality Objectives Process* (USEPA, 2006). The seven-step DQO process is as follows:

- Step 1 – State the Problem
- Step 2 – Identify the Goals of the Study
- Step 3 – Identify Information Inputs
- Step 4 – Define the Boundaries of the Study
- Step 5 – Develop the Analytic Approach
- Step 6 – Specify Performance or Acceptance Criteria
- Step 7 – Develop the Plan for Obtaining Data

11.1 Step 1 – State the Problem

Describe the Problem – The results of previous investigations conducted at SWMU 60 confirmed that the following COPCs are present at the SWMU at concentrations that may pose a potential risk to human health and the environment:

- Soil (Marina AST Area) – VOCs, PAHs, metals, and TPH GRO/DRO
- Soil (Suspected Debris Disposal Areas) – VOCs, PAHs, pesticides, and metals
- Groundwater – VOCs, PAHs, and metals
- Sediment – PAHs, pesticides, and metals

The nature and extent of contamination in surface and subsurface soil, groundwater, and sediment at the SWMU has not been delineated, and the limits of the suspected debris disposal areas have not been adequately investigated. Therefore, a Full RFI is warranted to collect additional data required to complete the characterization process and support the decision for Corrective Action Complete (CAC) or further action required.

Establish the Planning Team – The planning team includes the Navy, USEPA Region II, PREQB, and Baker. These team members are decision-makers for the DQO process.

Describe the Conceptual Site Model (CSM) – The CSM is described on Worksheet #10.

Identify Available Resources, Constraints, and Deadlines – The project team organization and project schedule are presented on Worksheets #5 and #16, respectively. The schedule presents the anticipated completion and/or submittal dates for specific tasks or documents.

11.2 Step 2 – Identify the Goals of the Study

The planning team's primary goal is to collect additional data required to complete the characterization process and support the decision for CAC or further action required. Specifically, data collected during the Full RFI will be of the quantity and quality necessary to provide technically sound and defensible answers to the following questions:

Marina AST Area

- What is the nature and extent of contamination in soil in the vicinity of the former underground piping system associated with the marina's ASTs?

- Is LNAPL present in the vicinity of the former underground piping system?

Suspected Debris Disposal Areas

- What are the boundary limits of the suspected debris disposal areas?
- Is the debris within the suspected disposal areas surficial in nature, or is buried debris present?
- What is the nature and extent of contamination in soil within/near the suspected debris disposal areas?

SWMU-Wide Groundwater

- What is the nature and extent of contamination in groundwater at the SWMU?
- Is groundwater suitable for use as potable water based on total dissolved solids (TDS) and total suspended solids (TSS) content and aquifer yield?
- What are the hydrogeologic conditions at the SWMU (e.g., groundwater flow direction, hydraulic gradient, hydraulic conductivity, groundwater flow velocity)?
- What is the interaction between shallow groundwater and Ensenada Honda?

Ensenada Honda

- What is the nature and extent of contamination in sediment from Ensenada Honda adjacent to the SWMU?

11.3 Step 3 – Identify Information Inputs

Existing information regarding the nature and extent of contamination and hydrogeologic conditions at the SWMU is limited. Additional data is required to adequately characterize the SWMU and determine the potential for future actions. The types of information required and sources used to resolve the decision statement presented in Step 2 include the following:

Types of Information Required	Sources of Information
Concentrations of COPCs (VOCs, PAHs, metals, and TPH GRO/DRO) in soil near the marina AST area	Existing and new analytical data from soil borings advanced at the SWMU
Concentrations of COPCs (VOCs, PAHs, pesticides, and metals) in soil within/near the suspected debris disposal areas	Existing and new analytical data from soil borings advanced at the SWMU
Boundary limits and depth (if buried) of debris disposal	Visual observations and data from the geophysical survey conducted within/near the suspected debris disposal areas
Concentrations of COPCs (VOCs, PAHs, and metals) and pesticides and TPH GRO/DRO in groundwater	Analytical data from existing and new monitoring wells installed at the SWMU
TDS and TSS content in groundwater	Analytical data from one of the existing wells (60SB04) and three of the new wells (60SB13 and two others TBD) installed at the SWMU
LNAPL thickness (if present)	Fluid level measurements from monitoring wells at the SWMU
Groundwater flow/hydrogeologic characteristics	Fluid level measurements from monitoring wells at the SWMU and adjacent SWMUs; slug test data from monitoring wells
Groundwater/Ensenada Honda interaction	
- Retaining pier/boat dock construction	Historical as-built drawings/interviews
- Salinity of groundwater	Salinity field measurements from monitoring wells at the SWMU
- Concurrent groundwater levels/Ensenada Honda water levels over a 72-hour period	Measurements collected using data loggers/pressure transducers
Concentrations of COPCs (PAHs, pesticides, and metals) and total organic carbon (TOC) content in sediment	Existing and new analytical data from sediment samples collected from Ensenada Honda adjacent to the SWMU

Note: Although pesticides and TPH GRO/DRO were not identified as COPCs in groundwater, they will be included in the analytical program due to their presence in soil.

The sampling design and rationale are provided on Worksheet #17, and the analytical specifications are provided on Worksheets #15 and #19. Worksheet #15 also lists the most stringent ecological and human health, risk-based Project Action Limits (PALs) and associated references. A complete set of ecological and human health, risk-based screening values and associated references are provided in Appendix A.

CompuChem will conduct the laboratory analysis for this project. The analytical methods shown on Worksheet #19 have been selected in an effort to attain, to the greatest extent practicable, limits of quantitation (LOQs), limits of detection (LODs), and detection limits (DLs) less than or equal to the most stringent PALs shown on Worksheet #15. However, based on the limitations of best available technology, the laboratory quantitation limits for some constituents still do not meet the associated PALs as shown on Worksheet #15.

The following constituents do not have established PALs for the respective media:

- Soil – idomethane and propionitrile
- Groundwater – idomethane

These constituents will be considered "absent" if they are not detected at the LOD. If these constituents are detected, their presence will be evaluated by factors such as frequency of detection, the maximum detected concentration, other constituents detected in the same area, etc.

For constituents that have a LOQ greater than the PAL but a LOD and DL less than the PAL, the analytical results for detected constituents will be reported to the DL. Non-detect constituents will be considered "absent" if they are not detected at the LOD.

For constituents that have a LOQ, LOD, and DL greater than the PAL, the analytical results for detected constituents will be reported to the DL. Non-detect constituents reported at the LOD will be further evaluated qualitatively in the human health and ecological risk assessments, which may be conducted as part of the CMS, or alternatively as part of this RFI if the site characterization is complete. An assessment of the associated uncertainty and impact to the overall estimates of risk and hazard will be provided, as appropriate, to determine if risk management decisions would be affected by the fact the LOD exceeds the PAL for a particular constituent(s).

11.4 Step 4 – Define the Boundaries of the Study

Figures 10 and 11 provide graphical representations of the spatial boundaries and sample locations. It should be noted that the specific number and locations of soil borings/monitoring wells within/near the suspected debris disposal areas will be based on results of the geophysical survey and contingent upon approval from the Navy, USEPA, and PREQB (see Worksheets #14 and #17).

It is anticipated that the field program will be implemented in the summer of 2012 and be completed within three weeks (see Worksheet #16). There are no significant physical barriers or access issues at the site at this time. Weather conditions, such as heavy rain/hurricanes, may raise a safety concern and can delay the field activities.

11.5 Step 5 – Develop the Analytic Approach

Individual constituents detected in the samples will be directly compared to the ecological and human health, risk-based screening values provided in Appendix A and appropriate basewide background values for metals (Upper Limit of the Means [ULM]) established in the *Revised Final II Summary Report for*

Environmental Background Concentrations of Inorganic Compounds (Baker, 2010). Comparison to the screening values will help to identify areas, contaminants, and conditions at the SWMU that may require further attention. If a given concentration at a given sampling point exceeds a screening value(s) and the background value (for metals only) for that constituent, then that sampling point will be considered to be impacted. Constituent concentrations above the screening values/background values will not automatically designate the SWMU as "dirty" or trigger a response action. However, exceeding a screening value/background value may suggest that further evaluation of the potential risks that may be posed by SWMU-related contaminants is appropriate. Further evaluation of constituent concentrations above screening values/background values will be conducted in the human health and ecological risk assessments, which may be conducted as part of the CMS, or alternatively as part of this RFI if the site characterization is complete.

11.6 Step 6 – Specify Performance or Acceptance Criteria

This step involves establishing the performance and acceptance criteria that the collected data will need to achieve in order to minimize the possibility of either making erroneous conclusions/decision errors or failing to keep uncertainty in estimates to within acceptable levels. A decision error occurs when the data misleads the planning team into choosing the wrong response action, in the sense that a different response action would have been chosen if the planning team had been able to access "perfect data" or absolute truth. The possibility of a decision error may occur as sampling design error and/or measurement error. Although the possibility of decision error can never be totally eliminated, it can be minimized and controlled. For this field program, sampling and measurement decision errors will be controlled by:

- Developing a sampling plan based on existing historical data biasing sample collection to known/perceived "hot spots/areas of concern" (i.e., locations within suspected/identified debris disposal areas) and locations outside those "hot spots/areas of concern" (i.e., downgradient/ sidegradient/upgradient of suspected/identified debris disposal areas and the former underground piping system associated with the marina's ASTs). By targeting known "hot spots/areas of concern" based on historical data, the sampling approach will ensure that worst-case site conditions (i.e., areas posing the greatest potential risk to human health and the environment) will be captured. In addition, targeting locations outside those "hot spots/areas of concern" will assist in characterization/delineation of potential contamination.

- Using qualified field personnel to conduct the sampling and data collection in accordance with procedures specified in this SAP

- Using analytical methods that achieve quantitation limits, to the extent practicable, less than the PALs. This controls measurement error by ensuring that the analytical techniques provide results that will allow for the determination of the absence of a constituent below the PALs. In addition, the proposed analytical methods are USEPA-approved methods with stringent QA/QC protocol. This minimizes error in the precision and accuracy/bias of the analytical program.

- Requesting Level IV data packages and subjecting the analytical results to independent, third-party data validation to identify laboratory discrepancies and minimize the use of erroneous data.

Although numerous efforts will be taken to control and minimize errors, there is always some uncertainty associated with sampling and analytical programs. Possible decision errors are as follows:

- Decision error based on a false positive (i.e., Type 1: Concluding there is risk present to human health or the environment, when in fact, there is no risk. Concluding that the extent of contamination is not defined, when in fact, it is.)

- Decision error based on a false negative (i.e., Type II: Concluding there is no risk present to human health or the environment, when in fact, there is risk. Concluding that the extent of contamination is defined, when in fact, it is not.)

Typically, ecological and human health risk assessments consider a Type 1 error rate, or significance level of 5% (or α=0.05) as acceptable. Risk management decisions will determine where the consequences of a false acceptance decision error are considered tolerable (gray region).

11.7 Step 7 – Develop the Plan for Obtaining Data

The sampling design and rationale are provided on Worksheet #17. Throughout the field program, sampling, analysis, and evaluation of data will be continually monitored by the Baker Task Manager and QA Officer to ensure that the proposed program is providing the input required in making the decisions noted above.

SAP Worksheet #12.1: Field Quality Control Samples

Measurement Performance Criteria Table – Soil

QC Sample	Analytical Group	Frequency	Data Quality Indicator (DQI)	Measurement Performance Criteria
Field Duplicate Samples	Appendix IX VOCs, LLPAHs, Pesticides, Metals, TPH GRO/DRO	10% for each group of primary environmental samples	Precision	<35% Relative Percent Difference (RPD)
Matrix Spike (MS)/Matrix Spike Duplicate (MSD) Samples		5% for each group of primary environmental samples	Matrix Interferences; Precision; Accuracy/Bias	<35% RPD
Field Blanks		One from each source of water used for decontamination	Accuracy/Bias-Contamination	No target constituents exceeding the LODs
Equipment Rinsate Blanks		One per day for reusable (non-dedicated/non-disposable) sampling equipment; one per batch of disposable sampling equipment	Accuracy/Bias-Contamination	No target constituents exceeding the LODs
Temperature Blanks		One per cooler	Accuracy/Representativeness	Samples received at the laboratory at 4° Celsius (C) (+/- 2° C)
Trip Blanks	Appendix IX VOCs, TPH GRO	One per cooler containing samples for volatile organics and/or TPH GRO analysis	Accuracy/Bias-Contamination	No target constituents exceeding the LODs

SAP Worksheet #12.2: Field Quality Control Samples

Measurement Performance Criteria Table – Groundwater

QC Sample	Analytical Group	Frequency	DQI	Measurement Performance Criteria
Field Duplicate Samples	Appendix IX VOCs, LLPAHs, Pesticides, Metals, TPH GRO/DRO	10% for each group of primary environmental samples	Precision	<20% RPD
MS/MSD Samples		5% for each group of primary environmental samples	Matrix Interferences; Precision; Accuracy/Bias	<20% RPD
Field Blanks		One from each source of water used for decontamination	Accuracy/Bias-Contamination	No target constituents exceeding the LODs
Equipment Rinsate Blanks		One per day for reusable (non-dedicated/non-disposable) sampling equipment; one per batch of disposable sampling equipment	Accuracy/Bias-Contamination	No target constituents exceeding the LODs
Temperature Blanks		One per cooler	Accuracy/Representativeness	Samples received at the laboratory at 4° C (+/- 2° C)
Trip Blanks	Appendix IX VOCs, TPH GRO	One per cooler containing samples for volatile organics and/or TPH GRO analysis	Accuracy/Bias-Contamination	No target constituents exceeding the LODs

SAP Worksheet #12.3: Field Quality Control Samples

Measurement Performance Criteria Table – Sediment

QC Sample	Analytical Group	Frequency	DQI	Measurement Performance Criteria
Field Duplicate Samples	Appendix IX LLPAHs, Pesticides, Metals	10% for each group of primary environmental samples	Precision	<35% RPD
MS/MSD Samples		5% for each group of primary environmental samples	Matrix Interferences; Precision; Accuracy/Bias	<35% RPD
Field Blanks		One from each source of water used for decontamination	Accuracy/Bias-Contamination	No target constituents exceeding the LODs
Equipment Rinsate Blanks		One per day for reusable (non-dedicated/non-disposable) sampling equipment; one per batch of disposable sampling equipment	Accuracy/Bias-Contamination	No target constituents exceeding the LODs
Temperature Blanks		One per cooler	Accuracy/Representativeness	Samples received at the laboratory at 4° C (+/- 2° C)

SAP Worksheet #13: Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (originating organization, report title and date)	Data Generator(s) (originating organization, data types, data generation / collection dates)	How Data Will Be Used	Limitations on Data Use
Soil and Groundwater Data	CH2M Hill: Site Characterization Report, MWR Marina, 12/1/1999	CH2M Hill: - Soil Data – BTEX and TPH GRO/ DRO - Groundwater Data – BTEX, PAHs, lead, and TPH GRO/DRO - Sample Collection Date: 5/5/1999-7/13/1999	- As a historical reference to provide an initial indication of the nature and extent of potential petroleum-related impacts attributed to the reported release from the former underground piping system associated with the marina's ASTs - To assist in developing the CSM (see Worksheet #10) - To focus the Full RFI	- Data used to generate the report were not validated - Insufficient data exists to determine the nature and extent of petroleum-related impacts - Data is outdated - No quantitative comparisons will be conducted using this data
Site History/ Background Information	NAVFAC Atlantic: Final Phase I/II Environmental Condition of Property Report, 7/15/2005	NAVFAC Atlantic: - Records Review - Aerial Photography Analysis - Interviews - Physical Site Inspection - Data Generation Date: 2004	- To provide site history/background information and gain an understanding of historical activities that may have led to a release(s) of solid and/or hazardous waste and hazardous constituents - To assist in developing the CSM (see Worksheet #10)	None
Soil, Groundwater, Surface Water, and Sediment Data		NAVFAC Atlantic: - Soil, Groundwater, Surface Water, and Sediment Data – VOCs, SVOCs, pesticides, herbicides, PCBs, and metals - Sample Collection Date: 5/7/2004-5/14/2004	- As a historical reference to provide an initial indication of the potential presence of contaminants that may be attributed to the suspected debris disposal areas - To assist in developing the CSM (see Worksheet #10) - To focus the Full RFI	- Data used to generate the report were not validated - Limited data set - Data is outdated - No quantitative comparisons will be conducted using this data
Soil, Groundwater, and Sediment Data	Baker: Final Phase I RCRA Facility Investigation Report, 7/22/2011	Baker: - Soil Data – VOCs, SVOCs/ LLPAHs, pesticides, metals, and TPH GRO/DRO - Groundwater Data – VOCs, SVOCs/LLPAHs, pesticides, metals, and TPH GRO/DRO - Sediment Data – SVOCs/LLPAHs, pesticides, and metals - Sample Collection Date: 1/12/2009-1/18/2009	- To evaluate the presence of existing contamination and identify COPCs - To assist in developing the CSM (see Worksheet #10) - To focus the Full RFI	- Suspected debris disposal areas have not been adequately investigated - Insufficient data exists to complete the characterization process and determine the nature and extent of contamination at the SWMU

SAP Worksheet #14: Summary of Project Tasks

This worksheet provides detailed activities associated with project initiation (section 14.1), sampling and data collection (Sections 14.2 through 14.4), laboratory analysis (Section 14.5), data validation/usability assessment (Section 14.6), data management and review (Section 14.7), and data evaluation and report preparation (Section 14.8). The sampling design and rationale are provided on Worksheet #17.

14.1 Project Initiation

Tasks associated with project initiation, including project planning/coordination, subcontractor procurement, mobilization/demobilization, utility clearance, and site clearing, are discussed below.

14.1.1 Project Planning/Coordination

Activities under this task will include project planning/coordination with the Navy, USEPA, and PREQB to ensure approval and completeness of this SAP. Prior to mobilization, a project orientation meeting with Baker personnel will be held to review the site history and layout, project objectives, information needs, field procedures, and health and safety issues presented in the site-specific HASP. In addition, the Navy will notify the USEPA and PREQB approximately two weeks in advance of the scheduled field activities in order to coordinate any agency field oversight.

This task also includes coordination with the Navy, USEPA, and PREQB regarding approval of the specific number and locations of borings/monitoring wells that are contingent upon results of the geophysical survey (see Section 14.2.2 and Worksheet #17). It is anticipated that this will include scheduling an on-site meeting and/or teleconference during the field program to review/discuss the results of the geophysical survey and proposed boring/well locations.

14.1.2 Subcontractor Procurement

Procurement activities under this task will consist of securing, scheduling, and coordination of the subcontractors required by Baker to complete the work associated with the field investigation described herein. It is anticipated that the following subcontractor services will be required:

- Site clearing and grubbing (RWEC)
- Geophysical surveying
- DPT sampling and monitoring well installation
- IDW transportation and disposal
- Surveying
- Laboratory analysis (CompuChem) including certification of the data by a Puerto Rico-certified chemist (Miriam Estrada, Analytical Environmental Services International, Inc.)
- Data validation (DataQual)

CompuChem and DataQual have already been procured for laboratory analysis and data validation services, respectively, since these services are an integral part of this SAP. In addition, RWEC routinely provides site clearing and grubbing services at NAPR. The subcontractors for geophysical surveying, DPT sampling and monitoring well installation, IDW transportation and disposal, and surveying services will be procured prior to implementing the field program.

14.1.3 Mobilization/Demobilization

This task will include pre-field preparation/coordination activities as well as mobilization and demobilization of Baker and subcontractor personnel/equipment. Pedro Ruiz (On-Island Navy Liaison) will be consulted during the mobilization efforts.

14.1.4 Utility Clearance

Prior to entering the field, base utility mapping will be reviewed to verify the presence/absence of subsurface utilities in the vicinity of the proposed boring/monitoring well locations. In addition, an electronic "shape" file that includes each proposed boring/well location will be obtained from the Computer Aided Design and Drafting (CADD)/Geographic Information System (GIS) at Baker and uploaded to a mapping-grade global positioning system (GPS) unit. Once in the field, the boring/well locations will be field-located using the GPS unit, and the presence/absence of subsurface utilities will be field-verified to the extent practicable. Some boring/well locations may need to be adjusted in the field to account for the presence of subsurface utilities or other below ground structures.

14.1.5 Site Clearing

Due to the presence of dense vegetation, site clearing activities will be required at some areas within the southeastern portion of the SWMU to open lines for the geophysical survey operations and provide access to the proposed boring/monitoring well locations. It is anticipated that the clearing activities will be conducted by RWEC of Naranjito, Puerto Rico using an ASV RC-100 skid steer equipped with a Magnum Systems, Inc. mulcher. Some areas may also be cleared by hand. The minimum area necessary for completion of the work will be cleared.

14.2 Field Investigation Procedures

The following sections provide guidance for the field investigation by describing detailed sampling and data collection procedures.

14.2.1 Field Verification of Wetland Boundary Delineation

The estuarine intertidal scrub-shrub broad-leaved evergreen (E2SS3) wetland and marine intertidal unconsolidated shore sand (M2US2) resource boundaries depicted on the figures associated with this SAP were delineated by Geo-Marine, Inc. in December 1999 based on 1993 color infrared and 1998 true color photography. These boundaries do not represent field-delineated, jurisdictional boundaries. Therefore, all wetland resource boundaries within the borders of the SWMU will be field-delineated and marked with wetland delineation flagging in accordance with the *Interim Regional Supplement to the Corps of Engineers Wetland Delineation Manual: Caribbean Islands Region* (U.S. Army Corps of Engineers [USACE], 2009).

Hydric soil and vegetation indicators, as well as hydrology indicators, will be evaluated as part of the delineation process. A hydric soil is defined as a soil that formed under conditions of saturation, flooding, or ponding long enough during the growing season to develop anaerobic conditions in the upper part (see Federal Register 59(133): 35680-35681, July 13, 1994). Hydric vegetation occurs in areas where inundation or soil saturation is either permanent or of sufficient frequency and duration to exert a controlling influence on the plant species present (USACE, 2009). The hydrology indicators evaluated will be those listed in the USACE document identified above including water marks (i.e., discoloration) and sediment deposits on the bark of woody vegetation or other fixed objects, water-stained leaves,

aquatic fauna (live individuals or dead remains), surface soil cracks that form when fine-grained or organic sediments dry and shrink, sparsely vegetated concave surfaces, and drainage patterns.

Data collected during the resource boundary delineations will be recorded in the field logbook(s) and on wetland determination data forms (Appendix B). Upon completion of the delineations, the boundaries will be surveyed for inclusion on site mapping. The survey will be conducted by a qualified subcontractor using conventional survey methods (see Section 14.3.4).

During implementation of the field sampling activities, potential wetland impacts will be minimized to the extent feasible. This may include, but not be limited to, clearing only the minimum area necessary for completion of the work and/or using hand tools for sample collection in lieu of heavy equipment. Because wetlands can contain soils with hydric characteristics, as well as sediment, the proposed boring/well locations within the boundary of the field-delineated wetland also will be evaluated to determine if they may support organisms (benthic macroinvertebrates such as fiddler crabs) typically associated with an estuarine aquatic community. If significant sediments are present at a boring location within the wetland, it will be noted in the field logbook(s), and the uppermost sample will be collected from 0 to 0.5 feet bgs (sediment depth interval) in lieu of 0 to 1 foot bgs (surface soil depth interval). In addition, TOC analysis will be added to the list of analytes for that sample.

14.2.2 Geophysical Survey

Electromagnetic (EM) terrain conductivity and magnetic surveys will be conducted at the three suspected debris disposal areas by a qualified subcontractor. EM instruments measure the electrical conductivity of subsurface materials by using a transmitter coil to generate an electromagnetic field that drives an electrical current into the ground. A receiver coil at a fixed separation from the transmitter is tuned and oriented to be insensitive to the transmitted field, and therefore measures only a secondary electromagnetic field generated by the subsurface electrical current. The strength and orientation of this field can be used to accurately estimate the bulk electrical conductivity (terrain conductivity) and relative ferrous and non-ferrous metallic content (in-phase response) of the surface/subsurface materials. Terrain conductivities are typically elevated in debris disposal areas due to the presence of exotic materials.

Magnetic surveys are sensitive to ferrous materials (e.g., iron or steel in tanks, drums, reinforcing, or debris) as well as materials that have been heated or burned at temperatures above their Curie point. Magnetometers measure the intensity of the earth's magnetic field along profiles or on a grid. In the absence of buried magnetic or magnetically susceptible objects (e.g., iron or steel containers, debris, burn residue, etc.), the intensity of the earth's magnetic field is generally constant or varies smoothly due to variations in lithologic composition at depth. However, magnetic objects may create localized/abrupt variations or anomalies in the magnetic field intensity.

The survey design and rationale are provided on Worksheet #17. The specific procedures will include the following:

1. EM terrain conductivity readings will be collected (to an approximate depth of 18 feet bgs depending on subsurface conditions) at 1-second (approximate 5-foot) intervals along transect profiles using a Geonics EM-31 MKII terrain conductivity meter (or equivalent). The transects will be oriented in the east-west direction and will be spaced at approximate 50-foot intervals. In addition, a transect oriented in the north-south direction will be scanned along the approximate centerline of each suspected disposal area. Readings will be digitally recorded using an Allegro Juniper data logger (or equivalent). Integrated location control (with sub-meter accuracy) will be provided by a differential global positioning system (DGPS) to ensure GIS-compatible mapping.

Each measurement location will be recorded using a mobile GPS receiver with radio-linked differential corrections applied relative to a fixed base station GPS receiver.

2. The EM terrain conductivity and in-phase data will be contoured in the field and overlain on site mapping. Footprints of any identified anomalies (e.g., ferrous and non-ferrous metallic debris) will be highlighted on the mapping.
3. A Geometrics cesium vapor magnetometer (MagMapper or equivalent) with two sensors will also be used (along the same profiles recorded by the EM survey) to collect magnetic field intensity measurements at two fixed heights. Magnetic data will be recorded digitally in the magnetometer's hard-wired memory and periodically transferred in the field to a portable computer for back-up and near real-time QC review.

4. A fixed position base station will be monitored continuously using a Geometrics proton precession magnetometer (or equivalent) at a location removed from any potential sources of magnetic interference. The base station data will be used to apply diurnal and atmospheric corrections.

5. Integrated location control (with sub-meter accuracy) will be provided by DGPS systems (as described above) to ensure GIS-compatible mapping.

6. The individual sensor data will be used to compute vertical, horizontal (north-south), and horizontal (east west) gradients suitable for determination of the analytic signal. The analytic signal converts the typical dipole (paired high-low) anomalies to single peak anomalies that better determine the source (e.g., debris) location.

7. The analytic signal magnetic data for each survey area will be processed and contoured/plotted in the field as a color overlay on site mapping. Footprints of any identified magnetic anomalies (e.g. tanks, drums, burn pits) will be highlighted on the map.

8. The GPS coordinates of ferrous and metallic debris will be recorded/mapped so that anomalies related to this debris can be discounted when the geophysical survey data is interpreted.

Where suspected anomalous targets are detected, numerous more closely spaced and variously oriented transects will be scanned to accurately determine the trace or footprint outline (and approximate depth where possible) of each target. The number, spacing, and orientation of additional transects will be determined in the field based on results from the initial/adjacent transects and professional judgment. The locations and depths (where possible) of any identified anomalies will be marked using wooden stakes and/or flagging and subsequently surveyed using conventional survey methods (in addition to the DGPS survey as described above) for inclusion on site mapping. The survey will be conducted by a qualified subcontractor (see Section 14.3.4). In addition, any surficial anomalies will be visibly inspected to identify the nature of the anomalies (e.g., type of anomaly/debris; condition; presence of liquids, sludges, solids in containers).

The geophysical surveying subcontractor will provide a report that documents the survey activities and results. The report will describe the geophysical procedures used and present figures depicting the locations of any identified anomalous targets. Back up information and associated interpretations of the data will also be provided as an appendix to the report.

Upon completion of the geophysical survey, the specific number and locations of proposed borings/monitoring wells within and near the suspected debris disposal areas will be discussed and agreed

upon with the Navy, USEPA, and PREQB. It is anticipated that this will be accomplished via an on-site meeting and/or teleconference during the field program.

14.2.3 Surface and Subsurface Soil Sampling

Surface and subsurface soil samples will be collected from a number of borings advanced by a qualified subcontractor using a Geoprobe® rig (or equivalent) and direct push methods. Soil cores will be collected from each boring in 4-foot increments from the ground surface to the desired depth using new, disposable acetate liners and Macro-Core® samplers (or equivalent). The sampling design and rationale, including the depth to which the borings will be advanced, are provided on Worksheet #17. Specific sample collection and field screening procedures to be used, as well as the criteria for selecting samples for laboratory analysis, are summarized below. Location-specific sampling method requirements are provided on Worksheets #18.1 and #18.2.

1. The sampling device (Macro-Core or equivalent) will be advanced to the desired depth.

2. Upon retrieval, the acetate liner will be removed from the sampler and sliced with a cutting tool to expose the soil core for field screening.

3. The soil core will be field-screened at approximate 6-inch intervals for total volatile organic vapors using a photoionization detector (PID). The highest measurement for each interval will be recorded in the field logbook(s).

4. The sample(s) for volatile organics analysis (e.g., VOCs, TPH GRO) will be collected using a TerraCore® sampling device (or equivalent). The sample(s) will be preserved in accordance with SW-846 Method 5035 by placing 5 gram aliquots of the sample in one pre-weighed, 40-milliliter, volatile organic analytes (VOA) vial containing methanol and two pre-weighed, 40-milliliter, VOA vials containing laboratory-grade deionized (DI) water (supplied with the TerraCore® sampling kit).

5. After collecting the sample(s) for volatile organics analysis, the remaining portion of the sample interval for non-volatile constituents (e.g., LLPAHs, pesticides, metals, TPH DRO) will be thoroughly homogenized in an aluminum pan using a new disposable, stainless steel spoon to ensure that the sample is as representative of the sample interval as possible.

6. The proper sample volumes will then be transferred into appropriate, laboratory-supplied containers (see Worksheet #19) and placed in a cooler with ice.

7. Pertinent sampling information, such as soil description (e.g., estimates of grain size, moisture content, discoloration, odor, other visual observations), PID measurements, sample number and location, sample depth, and time of sample collection, will be recorded in the field logbook(s).

Two soil samples will be collected for laboratory analysis from each boring as follows:

- One surface soil sample (0 to 1 foot bgs)
- One shallow subsurface soil sample (1 to 3 feet bgs)

No deeper, subsurface soil samples are proposed due to the occurrence of shallow groundwater. However, if groundwater is encountered at depths greater than 3 feet bgs, one deeper soil sample will be collected from each boring at a depth shallower than observed groundwater. Specifically, these deeper soil samples

will be collected from the depth interval exhibiting the highest PID measurement and/or visual/olfactory evidence of contamination. In lieu of any detected PID measurements or visual/olfactory evidence of contamination, the deeper soil samples will be collected based on lithologic location (i.e., from the soil/groundwater interface) and/or professional judgment. Consideration will be given to these deeper sample intervals to ensure that representative data is collected for use in the human health risk assessment (HHRA) that may be conducted as part of the CMS if warranted.

For boring locations in the wetland area, a determination will be made as to whether the sample is soil or sediment as previously described in Section 14.2.1. If significant sediments are present at a boring location, it will be noted in the field logbook(s), and the uppermost sample will be collected from 0 to 0.5 feet bgs (sediment depth interval) in lieu of 0 to 1 foot bgs (surface soil depth interval). In addition, TOC analysis will be added to the list of analytes for that sample.

Subsequent to sampling, the remaining soil will be placed back into each respective boring unless potential contamination is identified based on PID measurements and/or visual/olfactory observations or a monitoring well is installed. To the extent practicable, soils last out of the borehole will be returned first, thereby approximating the original stratigraphy. The remaining portion of the borehole, and borings in which potential contamination was identified, will be backfilled with bentonite grout. Contaminated soil will be managed as described in Section 14.3.2.

14.2.4 Monitoring Well Installation and Development

Monitoring wells will be installed at a number of borings used for soil sampling. The sampling design and rationale, including the anticipated depth to which the wells will be installed, are provided on Worksheet #17. The wells will be constructed, as directed by the Project Geologist, using 2-inch inside diameter (ID), Schedule 40, polyvinyl chloride (PVC) casing and screen materials with flush-joint threads. Well screens (0.010-inch slot) will be 10 feet in length and installed such that they intersect the groundwater table. It should be noted that often times the water table is very difficult to observe during drilling at NAPR, and little groundwater may be readily observed in the clay-rich fill material/fine-grained soils. In this case, the borehole(s) may be left open to identify the depth to which the water table equilibrates and ensure proper placement of the well screen(s). Specific well installation procedures are summarized below.

1. Upon completion of soil sampling as described in Section 14.2.3, the borehole will be reamed to the desired depth using the Geoprobe® rig (or equivalent) and 3 ¼-inch ID, hollow-stem augers. The well construction materials will be installed through the augers.

2. The well screen and bottom cap will be connected to threaded, flush-joint riser as the well string is lowered through the augers and set at the bottom of the borehole. An expandable, water-tight locking cap will be placed on the top of the riser.

3. The annular space around the well screen will be backfilled with a well-graded, fine to medium sand as the hollow-stem augers are being withdrawn from the borehole. The sand will extend to approximately 2 feet above the top of the screened interval. Due to the shallow occurrence of groundwater, a lesser distance above the top of the screened interval may be packed with sand to allow for placement of sealing material.

4. An approximate 2-foot thick sodium bentonite seal (minimum of 6 inches for very shallow wells) will be placed above the sand pack. If bentonite chips or pellets are used, they will be sized

appropriately, given the well and borehole diameter, and placed in a careful manner that will prevent bridging. The bentonite will be hydrated with potable water as necessary.

5. The annular space above the bentonite seal will be backfilled with cement/bentonite grout to prevent surface and near subsurface water from infiltrating into the screened groundwater monitoring zone. The grout will consist of 5 to 10% bentonite powder by dry weight and seven gallons of potable water per 94-pound bag of portland cement. For very shallow wells, the cement/bentonite grout may be omitted.

6. The depth intervals of all backfilled materials will be measured with a weighted measuring tape to the nearest 0.1-foot and recorded in the field logbook(s).

7. Wells in high traffic or manicured areas will be completed at the surface using a flush-mounted cover surrounded by a concrete pad. The concrete pad will be slightly elevated above ground surface and sloped away from the cover. Wells completed in areas that are heavily vegetated will have 2 to 3 feet of riser pipe "stickup" above ground surface. Steel, protective casing will be placed over the riser and surrounded by a concrete pad. The pad will be a minimum of 2 feet by 2 feet by 6 inches extending two feet bgs in the annular space around the well and set two inches into the ground elsewhere. If water table conditions prevent installation of a 2-foot thick bentonite seal, the concrete pad depth in the annular space around the well may be decreased. Steel bollards will be installed around the concrete pad as additional protection and painted a bright color to aid in visibility.

8. A lock will be placed on the expandable, water-tight cap or protective steel casing.

9. Pertinent well construction information, such as well depth, screened interval, and depth intervals of backfilled material, will be recorded in the field logbook(s).

Once a minimum of 24 hours has elapsed since installation, each new well will be developed using surging and bailing methods (or other suitable method) to remove fine-grained material and improve the hydraulic efficiency of the well. Water quality parameters (WQPs), including pH, oxidation/reduction potential (ORP), specific conductance, dissolved oxygen, temperature, and turbidity, will be measured throughout the development process. The wells will be developed until the following criteria are met:

- A minimum of three borehole volumes (plus the amount of any water added during the installation process) have been removed.

- The water is visually clear of sediments [e.g., turbidity is less than 10 Nephelometric Turbidity Units (NTUs)]

- Three successive WQP readings have stabilized within 0.2 Standard Units (S.U.) for pH, 20 millivolts for ORP, 3% for specific conductance, 10% for dissolved oxygen, and 10% for turbidity (when turbidity is greater than 10 NTUs). Although temperature readings will be recorded, they will not be used for stabilization evaluation. Temperatures measured at the surface are affected to some extent by the difference between ambient air and groundwater temperatures and thus can vary over short periods.

It should be noted that it may not always be possible to achieve turbidity measurements less than 10 NTUs due to the presence of clay-rich soils and fine-grained lithologies at NAPR. In the event turbidity measurements less than 10 NTUs cannot be achieved, turbidity will be evaluated based on three successive readings that have stabilized within 10% as indicated above.

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Wells installed as part of previous investigations will be evaluated to determine if re-development is warranted. Re-development will be warranted if 10% or more of the screen length has been filled in by sediment. This sediment matter can lead to turbid samples and possibly incorrect conclusions about groundwater analytical results. If re-development is warranted, the procedures described above will be followed. However, additional time and volume of water removed may be required to remove the sediment from the well. The well development information will be recorded in the field logbook(s).

14.2.5 Monitoring Well Abandonment and Replacement

Two of the existing monitoring wells (60SB04 and 60SB05) installed during the Phase I RFI will be abandoned since the screened intervals are below the water table and the wells are not suitable to assess the presence/absence of LNAPL. Specific abandonment procedures are summarized below:

- The concrete pad around each well will be demolished and the flush-mounted cover removed.
- The well casing/screen and annular materials will be over drilled using a Geoprobe® rig (or equivalent) and 3 ¼-inch ID, hollow-stem augers. Alternatively, the well string may be pulled from the ground without over drilling.
- The borehole will then be backfilled with bentonite grout and capped with concrete at the surface.

Upon completion of the well abandonment, replacement wells will be installed adjacent to (within 5 to 10 feet) the initial wells. The replacement wells will be installed/developed using methods described in Section 14.2.4 and given an “R” designation (e.g., 60SB04R).

If any of the remaining existing wells (MW-1, MW-4, 6E-01, 6E-02, and 6SB-02) cannot be located or have been damaged (i.e., well casing broken off), a decision will be made as to whether the well(s) needs replaced or can be repaired. The decision to replace a well will be based on the well’s function in the monitoring network and whether sufficient data needs can be obtained from other wells in the network.

14.2.6 Groundwater Sampling

Groundwater samples will be collected from new monitoring wells and existing wells after completion of well development and the water level has returned to equilibrium. A reasonable effort will be made to conduct the sampling after a minimum of 14 days has elapsed since completion of well development. However, this will be dependent on whether subsequent field activities are being conducted by Baker at other SWMUs/Areas of Concern (AOCs). In any case, the sampling will be planned accordingly such that the maximum time period between completion of well development and sampling is allowed. The sampling design and rationale are provided on Worksheet #17.

The groundwater samples will be collected using decontaminated, stainless steel bladder pumps and low flow purging/sampling methods to minimize sampling-induced turbidity problems and provide samples representative of ambient groundwater quality. New Teflon® bladders and new Teflon®-lined polyethylene tubing will be used for each well. The pump intake will be placed at the depth of any distinct, more permeable/productive water-bearing zone identified during well installation. If no distinct, more permeable/productive water-bearing zone is discernable, the pump intake will be placed within the bottom half of the screened interval at least 2 feet from the bottom of the well. Specific groundwater sampling procedures are presented in *Ground Water Sampling Procedure, Low Stress (Low Flow)*

Purging and Sampling (USEPA, 1998) (Appendix C) and summarized below. Location-specific sampling method requirements are provided on Worksheet #18.3.

1. The pump and tubing will be slowly lowered into the well to the desired depth and connected to the controller and compressor. Care will be taken during placement of the pump to minimize disturbance in the water column and dislodging of any sediment that has accumulated at the bottom of the well.

2. Before starting the pump, the static water level in the well will be measured. The water level indicator will be left in the well just above the water.

3. The well will be pumped at a rate of approximately 200 to 500 milliliters per minute (ml/min), and the water level will be measured at approximate five-minute intervals. Flow measurement calculations will be conducted using a graduated container and a stopwatch.

4. The pumping rate will be adjusted, if needed, to ensure a steady, low flow that results in a stabilized water level (drawdown of 0.3 feet or less). It should be noted that this is dependent on site-specific hydrogeology, and a minimal drawdown of 0.3 feet or less may be difficult to achieve under some circumstances due to fine-grained lithologies and geologic heterogeneity.

5. WQPs, including pH, ORP, specific conductance, dissolved oxygen, temperature, and turbidity, will be measured at approximate 5-minute intervals during purging and recorded on groundwater sampling forms (Appendix B). The field testing will be conducted within a flow-through cell that limits exposure of the groundwater to the atmosphere while the field measurements are recorded. Salinity measurements will also be collected during purging and used when evaluating the interaction between shallow groundwater and Ensenada Honda (see Section 14.2.11).

6. Well purging will be considered complete when three successive WQP readings have stabilized within 0.1 S.U. for pH, 10 millivolts for ORP, 3% for specific conductance, and 10% for dissolved oxygen and turbidity. Temperature readings will be recorded, but not used for stabilization evaluation. Temperatures measured at the surface are affected to some extent by the difference between ambient air and groundwater temperatures and thus can vary over short periods. If key WQPs (dissolved oxygen and turbidity) have not reached a stable plateau after four hours of purging, purging may be discontinued and the sample collected. A full explanation of attempts to achieve stabilization will be recorded in the field logbook(s).

7. If a well exhibits insufficient yield (i.e., purges dry), purging will be discontinued. Sampling will commence as soon as the volume in the well has recovered sufficiently to allow collection of the sample.

8. Upon WQP stabilization (or sufficient recharge), the sample will be collected from the end of the tubing at a flow rate between 100 and 250 ml/min and placed into appropriate, laboratory-supplied containers with the appropriate preservatives (see Worksheet #19). Samples for dissolved metals will be filtered in the field prior to preservation using 0.45-micron disposable filters.

14.2.7 Hydraulic Conductivity Testing

Rising and/or falling head slug tests will be conducted at each new monitoring well after a minimum of 24 hours has elapsed from completion of sampling and the water level has returned to equilibrium. In addition, slug tests will be conducted at two of the existing wells (MW-1 and MW-4). Slug tests will not

be conducted at the remaining three existing wells (6E-01, 6E-02, and 60SB02) since they were constructed as "temporary wells". The slug tests will be conducted using a computerized data logger/pressure transducer. Prior to slug testing, the water level will be measured in the well. The data logger/pressure transducer will then be placed near the bottom of the well, and the water level will be allowed to equilibrate. The data logger will be started to ensure collection of data at the earliest possible time. A decontaminated slug (solid cylinder) will then be introduced into the well to displace an initial volume of water. The water-bearing zone's response to change in static conditions will be measured using the data logger/pressure transducer (falling head slug test). Once the water level returns to static conditions, the slug will be removed, and the response to change in static conditions will again be measured (rising head slug test). When possible, multiple tests will be conducted at a well to provide comparative data.

Upon completion of the slug tests, the data will be downloaded and processed using Microsoft Excel. Aqtesolv® Pro 4.0 will be used to facilitate the data analysis using an appropriate solution (e.g., Bouwer and Rice, Hvorslev) based on site-specific geologic/hydrogeologic conditions. Although falling head slug test data may be recorded at wells in which the water level is within the screened interval, these data will not be used in the hydraulic conductivity estimates to eliminate any influence from the sand pack in the curve match solution.

14.2.8 Fluid Level Measurements

Fluid level measurements will be collected from each new monitoring well shortly after installation and prior to and after well development, sampling, and hydraulic conductivity testing activities. In addition, prior to groundwater sampling, a synoptic set of fluid level measurements will be collected from the new and existing wells at SWMU 60 and a number of existing wells at adjacent SWMUs/AOCs (e.g., JP-5 Hill and Diesel Fuel Marine Area of SWMU 74, AOC F 1995) to more accurately interpret the groundwater flow regime and any LNAPL thickness if present.

The fluid levels will be measured using a water level indicator or oil/water interface probe as appropriate. The depth to fluid level from the surveyed reference point on top of each PVC well riser will be recorded to the nearest 0.01 foot in the field logbook(s).

14.2.9 Sediment Sampling

Sediment samples will be collected from open water areas in Ensenada Honda. The sampling design and rationale are provided on Worksheet #17. Location-specific sampling method requirements are provided on Worksheet #18.4.

It is anticipated that samples closer to the shoreline can be collected by wading depending on the water depth. However, some samples likely will require the use of a boat. The samples will be collected from 0 to 0.5 feet bgs using a decontaminated ponar dredge or sediment corer. The ponar dredge consists of a clam shell arrangement of two buckets that close upon impact with the bottom. The sampler is attached to a rope and lowered through the water column. A screen on the top of the sample compartment permits water to pass through the sampler as it descends thus reducing the "shock wave" and permits direct access to the secured sample without opening the closed jaws. The sediment corer consists of a barrel sampler with disposal acetate liners (similar to the Geoprobe® Macro-Core sampler) and extension rods for sampling through various water column depths. The sampler is lowered through the water column and manually pushed into the sediment to the desired depth.

Upon retrieval, the samples will be handled as described in Section 14.2.3. Pertinent sampling information, such as sediment description (e.g., color and texture), presence or absence of aquatic invertebrates, sample number and location, sample depth, and time of sample collection, will be recorded in the field logbook(s).

14.2.10 Field QA/QC Sampling

Field QA/QC samples will be collected to assess the precision and accuracy/bias of the data and include field duplicate samples, MS/MSD samples, trip blanks, field blanks, and equipment rinsate blanks as described below. Although the MS/MSD is not typically considered a field QA/QC sample, it is included herein because location determination is often established in the field. The field QA/QC sample measurement performance criteria and sample summary are presented on Worksheets #12 and #20, respectively.

Field Duplicate Samples – Field duplicate samples will be collected at a minimum frequency of 10% for each group of primary environmental samples of a similar matrix. The duplicate samples will consist of one unique sample, split into two aliquots, and analyzed independently for the same parameters as the corresponding original samples. Duplicate soil samples analyzed for parameters other than VOCs and TPH GRO will be homogenized and split. Samples for VOC and TPH GRO analyses will not be homogenized, but select segments of the soil will be collected. Duplicate water samples will be collected immediately after the corresponding primary sample for each analytical suite. The results will be used to evaluate the consistency and reproducibility of the field sampling and analytical procedures (i.e., precision).

MS/MSD Samples – MS/MSD samples will be collected at a minimum frequency of 5% for each group of primary environmental samples of a similar matrix. The samples will be collected in the field using the same procedures as duplicate samples and analyzed independently for the same parameters as the corresponding original samples. The results will be used to evaluate analytical bias and precision for specific constituents in specific sample matrices.

Trip Blanks – Trip blanks are samples of analyte-free water prepared at the laboratory before commencement of the sampling event and shipped to the sampling team along with the unopened sample containers. The trip blanks will be randomly selected and included in each cooler containing samples for

volatile organics analysis. The results will be used to evaluate potential field or laboratory contamination introduced during sampling, storage, and transport (i.e., accuracy/bias).

Field Blanks – One field blank will be collected from each source of water used for decontamination purposes. The field blanks will be collected under representative field conditions and analyzed for the same parameters as the corresponding environmental samples. It is anticipated that two different sources of water (i.e., store-bought distilled water and laboratory-grade DI water) will be used for this investigation. The results will be used to evaluate potential field or laboratory contamination introduced during sampling, storage, and transport (i.e., accuracy/bias).

Equipment Rinsate Blanks – One equipment rinsate blank will be collected each day for reusable (non-dedicated/non-disposable) sampling equipment. In addition, one equipment rinsate blank will be collected per batch of disposable sampling equipment. The equipment rinsate blanks will be collected under representative field conditions by running laboratory-grade DI water over/through the sampling equipment and placing it into the appropriate sample containers for laboratory analysis. The samples will be analyzed for the same parameters as the corresponding primary environmental samples. The results from the equipment rinsate blanks will be used to verify that the sampling equipment did not contribute to contamination of the samples (i.e., accuracy/bias).

14.2.11 Shallow Groundwater/Ensenada Honda Interaction

The interaction (i.e., tidal influence) between shallow groundwater and Ensenada Honda will be evaluated to provide a more comprehensive understanding of the hydrogeologic conditions at the SWMU. As part of this evaluation, readily available information regarding construction of the retaining pier/boat docks (e.g., construction material, depth of penetration bgs) will be obtained through review of as-built drawings (if available) and interviews of NAPR personnel. In addition, salinity measurements will be collected from the new and existing monitoring wells at the SWMU during groundwater sampling as described in Section 14.2.6.

Concurrent water levels will also be collected from Ensenada Honda, monitoring wells MW-4 and 60SB13, one of the new wells (TBD) located near the southwestern portion of the SWMU, and one of the new wells (TBD) located near the central or eastern portion of the SWMU. The water levels will be collected over a 72-hour period using computerized data loggers/pressure transducers.

Prior to the test, a stand pipe (60SP01 as shown on Figure 10) will be installed in Ensenada Honda adjacent to the boat dock near well 60SB13. The stand pipe will be constructed by connecting a 10-foot section of 2-inch ID, Schedule 40, 0.010-inch slot well screen with a bottom cap to threaded, flush-joint riser of the appropriate length. The stand pipe will be lowered through the water column and manually pushed into the sediment approximately 6 to 8 inches for stability purposes. For additional stability, the stand pipe will be secured to the boat dock.

The data loggers/pressure transducers will be placed near the bottom of the stand pipe/wells and programmed/synchronized to collect water level measurements every 30 minutes over the 72-hour test period. Upon completion of the test, the data will be downloaded and processed using Microsoft Excel.

14.3 Other Investigation Considerations

Other investigation considerations, including equipment decontamination, IDW management/disposal, sample preservation, handling, and management, and surveying are discussed below.

14.3.1 Equipment Decontamination

The Geoprobe® rig and associated sampling tools will be decontaminated using a steam cleaner or pressure washer prior to starting any work and immediately following completion of the field program. At a minimum, the Geoprobe® rods and Macro-Core samplers will be decontaminated between borings by washing the equipment in potable water and Liquinox® detergent followed by a distilled water rinse.

Disposable sampling equipment will be used to the extent practicable in order to minimize the potential for cross-contamination. Non-disposable sampling equipment (other than the bladder pumps used for groundwater sampling) that comes in direct contact with the sample (e.g., Macro-Core drive shoes, ponar dredge, slugs) will be decontaminated between each sample as follows:

1. Wash equipment thoroughly with Liquinox® detergent (or equivalent) and potable water using a brush to remove any particulate matter or surface film
2. Rinse equipment thoroughly with potable water
3. Rinse equipment with 10% nitric acid solution
4. Rinse equipment thoroughly with distilled water
5. Rinse equipment with methanol
6. Rinse equipment thoroughly with distilled water

Prior to groundwater sampling each day, the bladder pumps will be decontaminated as follows:

1. Dismantle pump and wash components thoroughly with Liquinox® detergent (or equivalent) and potable water
2. Rinse components thoroughly with potable water
3. Rinse components with 10% nitric acid solution
4. Rinse components thoroughly with distilled water
5. Rinse components with methanol
6. Rinse components thoroughly with distilled water

Between wells, the bladder pumps will be dismantled, and the components will be washed thoroughly with Liquinox® detergent (or equivalent) and potable water followed by a distilled water rinse. New Teflon® bladders and new Teflon®-lined polyethylene tubing will be used for each well.

Heavy equipment (e.g., hollow-stem augers) will be decontaminated between borings/monitoring wells at a central decontamination pad using a steam cleaner or pressure washer. Decontamination fluids will be pumped regularly from the pad into Department of Transportation-approved 55-gallon drums.

14.3.2 IDW Management/Disposal

IDW will consist of potentially contaminated soil, groundwater, and decontamination fluids. Soil will be placed back into each respective boring unless potential contamination is identified based on PID measurements and/or visual/olfactory observations or a monitoring well is installed. To the extent practicable, soils last out of the borehole will be returned first, thereby approximating the original stratigraphy. Potentially contaminated soil/soil from monitoring well borings and groundwater/ decontamination fluids will be containerized separately in Department of Transportation-approved 55-gallon drums. The drums will be properly labeled and temporarily staged at a secure location on base until proper disposal arrangements are made.

Upon completion of the field program, the IDW will be sampled and characterized for disposal. The analytical parameters presented herein were selected based on those being investigated at the SWMU and disposal facility requirements. One composite sample will be collected from the soil IDW and analyzed for Toxicity Characteristic Leaching Procedure (TCLP) VOCs, pesticides, and metals, Appendix IX LLPAHs, TPH GRO and DRO, and RCRA hazardous waste characteristics including ignitability, reactivity, and corrosivity. The sample will be compiled from individual, discrete (grab) samples of equal volume collected from each of the 55-gallon drums containing soil IDW. The individual, discrete samples for parameters other than VOCs and TPH GRO will be thoroughly homogenized in an aluminum pan using a new disposable, stainless steel spoon. The proper sample volumes will then be transferred into appropriate, laboratory-supplied containers (see Worksheet #19) and placed in a cooler with ice. The sample for VOC and TPH GRO analyses will not be homogenized, but select segments of the soil will be collected.

One composite sample will also be collected from the liquid IDW and analyzed for Appendix IX VOCs, LLPAHs, pesticides, and metals (total fraction), TPH GRO and DRO, and the aforementioned RCRA hazardous waste characteristics. The sample will be compiled from individual, discrete (grab) samples of equal volume collected from each of the 55-gallon drums containing liquid IDW. The individual, discrete samples will be homogenized in a clean container. The proper sample volumes will then be transferred into appropriate, laboratory-supplied containers (see Worksheet #19) and placed in a cooler with ice.

The drums of IDW will be removed and properly disposed of by a qualified subcontractor upon receipt and review of the IDW analytical data.

14.3.3 Sample Preservation, Handling, and Management

Sample preservation, handling, and management procedures are important components to maintaining sample integrity. Samples for laboratory analysis will be place into appropriate sample containers with the appropriate preservatives and maintained at 4° C (+/-2° C) during storage and shipment. The sample containers will be provided by CompuChem. The specific sample containers, sample volumes, preservation requirements, and holding times are provided on Worksheet #19.

Each sample will be designated with a unique number, and each container will be identified using a standard label taped to the container to ensure it does not separate. Chain-of-custody procedures will be followed throughout the sampling program to ensure a documented, traceable link between measurement results and the sample or parameter they represent. These procedures are intended to provide a legally acceptable record of sample collection, identification, preparation, storage, shipping, and analysis. Proper chain-of-custody documentation will be maintained for all samples from the time of collection until they are shipped to the laboratory. Specific sample labeling and custody procedures are provided on Worksheet #27.

The samples will be properly packaged in coolers and delivered by Federal Express (priority overnight) to CompuChem in Cary, North Carolina for arrival before 10:00 a.m. the following day. At least one member of the field team may also remain on the island until receipt of all shipments by the laboratory has been verified. This will minimize any potential re-sampling costs associated with mobilization. Tracking numbers for each shipment will be forwarded to the Data Management Specialist to assist in verification of receipt of samples by the laboratory.

Sample management activities will involve coordination with the laboratory, sample and analyses tracking, and submittal of the data to the independent, third-party data validator (DataQual). Sample management will also involve resolving potential problems (re-analysis, electronic deliverable formatting, and other technical information) between Baker, the laboratory, and the data validator.

14.3.4 Surveying

The sediment sample locations will be surveyed for horizontal position by Baker using a mapping-grade GPS unit. The horizontal data will be within approximately 2 feet and referenced to the U.S. State Plane coordinate system, Puerto Rico/Virgin Island 5200, North American Datum (NAD) 1983.

Upon completion of the field program, the field-delineated wetland boundaries, boundary limits of any identified debris disposal areas, boring locations, and monitoring well locations will be surveyed by a qualified subcontractor using conventional survey methods. The locations will be surveyed for topographic elevation and horizontal position. The vertical data will be to the nearest 0.01-foot and referenced to the mean low water plus 100.00 feet as established by the U.S. Navy Survey Section (November 1941). The horizontal data will be to the nearest 0.05-foot and referenced to the U.S. State Plane coordinate system, Puerto Rico/Virgin Island 5200, NAD 1983.

14.4 Documentation and Field Reporting Requirements

Specific procedures associated with documentation and field reporting requirements are discussed below.

14.4.1 Field Logbooks

Field logbooks, wetland determination data forms, equipment calibration forms, and groundwater sampling forms will be used to record sampling activities and other detailed information specific to sampling and data collection. Example field data collection forms are included in Appendix B. Each member of the field team will maintain a logbook, which documents their field activities. The FOL will also keep a separate logbook, which contains a daily summary of activities including references to individual field logbooks.

Daily entries into the logbook will contain a variety of information pertaining to the work such that the site activities may be reconstructed. At the beginning of each day, information including date, start time, weather, and field personnel present (including subcontractors) will be recorded by each team member. During the day, an ongoing record of all pertinent site activities will be recorded in the logbook. Specific information recorded in the logbook will include, but not be limited to, the following:

- Soil boring and monitoring well construction information
- Sample collection methods, sample numbers, media, analysis, and time
- Any problems encountered
- Deviations from the SAP and justification
- Field measurements
- Equipment used
- Equipment decontamination procedures
- Photograph log
- IDW Drum log
- Other pertinent information

All entries will be made in indelible ink. Field logbook pages will be dated and sequentially numbered. In addition, the last page of each daily entry will be signed or initialed by the responsible field personnel completing the log. No erasures will be permitted. If an incorrect entry is made, the entry will be crossed out with a single line, initialed, and dated. Any unused portion of a page will be struck through and initialed by the responsible field personnel.

14.4.2 Field Reporting Requirements

The FOL will prepare daily field progress reports for each day's field activities. The following information, at a minimum, will be included in the reports:

- Baker and subcontractor personnel on site
- Major activities of the day
- Samples collected
- Problems encountered
- Other pertinent site information

The field progress reports will be e-mailed to the Baker Project Manager, QA Officer, Task Manager, and Data Management Specialist upon completion of work each day.

14.5 Laboratory Analysis

Samples collected for laboratory analysis will be submitted to CompuChem in Cary, North Carolina. CompuChem will calibrate, maintain, test, and inspect the instruments used for analysis of the samples as indicated on Worksheets #24 and #25. In addition, CompuChem will process, prepare, and analyze the samples in accordance with the analytical standard operating procedures (SOPs) listed on Worksheet #23 and included in Appendix D. CompuChem's Quality Manual is also included in Appendix D. Analytical results for soil and sediment will be reported on a dry-weight basis.

Specific constituents to be included in each analytical fraction and identification of the analytical methods to be used are provided on Worksheets #15 and #19, respectively. Worksheet #15 also summarizes the most stringent PALs and quantitation goals for the target parameters and chosen analytical methods. The laboratory analytical methods shown on Worksheet #19 were selected in an effort to attain, to the greatest extent practicable, LOQs, LODs, and DLs that are less than the PALs. However, based on the limitations of the best available analytical technology, the laboratory quantitation limits for some constituents still do not meet the associated PALs. An explanation of how these will be addressed is provided on Worksheet #11.

The data will be certified by a Puerto Rico-certified chemist (Miriam Estrada, Analytical Environmental Services International, Inc.). Complete Level IV data packages will be provided by the laboratory for each sample delivery group (SDG) within 28 calendar days from receipt of the samples. Raw data included in the package will be sufficient to recalculate concentrations. Analytical results will also be provided in an electronic data deliverable (EDD) compatible with Windows applications. Additional information regarding requirements for the data package deliverables are provided on Worksheet #30.

14.6 Data Validation/Usability Assessment

Independent, third-party data validation will be conducted by DataQual of St. Louis, Missouri. The laboratory analytical data (except solid and liquid IDW data) will be evaluated to assess the technical adequacy and usability of the data. The data will be validated in accordance with the SW-846 methods utilized by the laboratory, specifications set forth in the USEPA Region II SOPs for Validation of Organic Data Acquired using SW-846 Methods, and professional judgment. It should be noted that Region II has not developed a validation checklist SOP for the methods used to assess TPH GRO and DRO (SW-846 Method 8015C) and inorganics (SW-846 Methods 6010C/6020A/7470A/7471B). Therefore, alternative worksheets will be used. Non-compliant analytical results will be qualified using Region II flagging conventions. Specific data verification and validation requirements are provided on Worksheets #34 through #36. The data validation worksheets are included in Appendix E.

Data validation reports will be prepared for each SDG. In general, the reports will include, but not be limited to, the following:

- Introduction which includes validator name, report date, project name, laboratory name, SDG number, sample identifications, matrix, and type of analysis
- Overall evaluation of the data and potential usability issues
- Specific evaluation of the data with respect to data completeness, technical holding times, initial/continuing calibration, blanks, internal standards, surrogates, MS/MSDs, serial dilutions, field duplicates, and identification/quantitation
- Summary of data qualifications
- Glossary of qualification flags and abbreviations
- Copies of analytical result pages marked with the appropriate changes in results/qualifiers based on the data validation findings; each analytical result page with changes will be initialed and dated and still included if there are no changes in results/qualifiers

Upon completion of the validation process, the data will be evaluated for its intended use based on review of the data validation report, qualifications to the data, performance criteria, and any deviations from planned activities. The usability assessment is discussed in more detail on Worksheet #37.

14.7 Data Management and Review

The Baker Data Management Specialist will manage sample tracking and assimilation of the data from field collection through analysis, validation, and upload to the project database. As previously discussed, the laboratory will provide Level IV data packages and EDDs for each SDG. The data validator will be required to add any qualifiers to both versions. Analytical data will be checked to ensure the values and any qualifiers are appropriately transferred to the EDDs. These checks will include comparison of the

hardcopy data and qualifiers to the EDD. Once the data has been uploaded to the project database, another check will be conducted to ensure the results were loaded accurately.

Data management activities will also consist of entering field-generated data, including fluid level measurements and water quality measurements, onto computerized spreadsheets. Checks will be performed against the field logbooks/data collection forms to ensure completeness and accuracy.

14.8 Data Evaluation and Report Preparation

This task involves efforts related to the data once it is received from the laboratory and is validated. In addition, this task involves evaluation of field-generated data. In general, the data evaluation activities will include, but not be limited to, the following:

- Tabulation of field and analytical data
- Comparison of analytical data to the ecological and human health, risk-based screening values provided in Appendix A and appropriate basewide background values for metals (ULM) established in the *Revised Final II Summary Report for Environmental Background Concentrations of Inorganic Compounds* (Baker, 2010).
- Generation of test boring and well construction records
- Generation of a groundwater table elevation contour map and other diagrams/figures associated with field notes or data received from the laboratory (e.g., sampling location maps, contaminant distribution maps)
- Evaluation and interpretation of geologic, hydrogeologic, field, and analytical data

Upon evaluation of the data, a Full RFI report will be prepared. In general, the report will include, but not be limited to, the following:

- Introduction – This section will provide an introduction and include the regulatory framework, purpose of the report, objectives of the Full RFI, and report organization.
- Background – This section will include a description and history of NAPR and SWMU 60. In addition, this section will present a summary of results from previous investigations conducted at the SWMU.
- Physical Characteristics of Study Area – This section will provide the environmental setting, including the regional geology and hydrogeology. Regional and local climatic conditions that may be relevant to the environmental impacts of contaminated media at SWMU 60 will also be discussed as warranted.
- Full RFI Activities – This section will describe the basis for the Full RFI and will include a description of sample locations, sample collection and handling procedures, QA/QC procedures, and analytical methods used. In addition, this section will discuss any problems encountered, including deviations from this SAP, and problem resolution.

- Physical Results – This section will present the current site conditions at SWMU 60 at the time of the Full RFI. The SWMU-specific geology and hydrogeology, as ascertained from the soil boring/monitoring well installation program and other field observations, will also be discussed.

- Analytical Results – This section will present the analytical results and an interpretation of the nature and extent of contaminants present in soil, groundwater, and sediment at the SWMU. In addition, this section will present a summary of the data validation/usability assessment.

- Site Conceptual Model – Information from the physical and analytical results (nature and extent of contamination), as well current site conditions, will be used to update the CSM.

- Conclusions and Recommendations – Information from the physical and analytical results (nature and extent of contamination) will be synthesized into conclusions regarding SWMU conditions. Recommendations will be made based on these conclusions as to whether a CMS is warranted or the SWMU can proceed toward CAC. An ecological risk assessment (ERA) and HHRA may be conducted as part of the CMS. Alternatively, the ERA and HHRA may be conducted as part of the Full RFI if the site characterization is complete.

- References – Source material used in development of the Full RFI Report will be documented in the References section of the report.

The Full RFI report will be posted to the NAPR web site under the document library. In addition, the data obtained during the field program will be incorporated into the web-based GIS system currently residing on the NAPR website. Baker will also provide updates of current activities associated with this project in the RCRA Quarterly Progress Reports for NAPR.

SAP Worksheet #15.1: Reference Limits and Evaluation Tables

Matrix: Soil

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; pH

Analyte	CAS No.	PAL (µg/kg)	PAL Reference	Quantitation Goal (µg/kg)	Laboratory-Specific Limits (µg/kg)		
					LOQs	LODs	DLs
Appendix IX VOCs (8260B)							
1,1,1,2-Tetrachloroethane	630-20-6	100	Eco – CCME 2007	100	5.0	1.0	0.32
1,1,1-Trichloroethane	71-55-6	100	Eco – CCME 2007	100	5.0	1.0	0.50
1,1,2-Trichloroethane	79-00-5	100	Eco – CCME 2007	100	5.0	2.5	0.72
1,1-Dichloroethane	75-34-3	100	Eco – CCME 2007	100	5.0	1.0	0.47
1,1-Dichloroethene	75-35-4	100	Eco – CMME 2007	100	5.0	2.5	1.1
1,2,3-Trichloropropane	96-18-4	5.00	HH – USEPA RSL	5.00	5.0	2.5	0.91
1,2-Dibromo-3-chloropropane	96-12-8	5.40	HH – USEPA RSL	5.40	5.0	2.5	0.65
1,2-Dibromoethane	106-93-4	34.0	HH – USEPA RSL	34.0	5.0	0.25	0.22
1,2-Dichloroethane	107-06-2	402	Eco – MHSPE 2000	402	5.0	1.0	0.27
1,2-Dichloropropane	78-87-5	940	HH – USEPA RSL	940	5.0	2.5	0.63
2-Butanone	78-93-6	2,800,000	HH – USEPA RSL	2,800,000	12	2.5	1.3
2-Hexanone	591-78-6	21,000	HH – USEPA RSL	21,000	12	2.5	1.0
3-Chloro-1-propene	107-05-1	680	HH – USEPA RSL	680	5.0	2.5	1.1
4-Methyl-2-pentanone	108-10-1	530,000	HH – USEPA RSL	530,000	12	2.5	1.3
Acetone	67-64-1	6,100,000	HH – USEPA RSL	6,100,000	12	6.3	5.0
Acetonitrile	75-05-8	87,000	HH – USEPA RSL	87,000	5.0	2.5	0.92
Acrolein	107-02-8	15.0	HH – USEPA RSL	15.0	**50**	10	5.7
Acrylonitrile	107-13-1	240	HH – USEPA RSL	240	50	10	3.7
Benzene	71-43-2	101	Eco – MHSPE 2000	101	5.0	1.0	0.31
Bromodichloromethane	75-27-4	270	HH – USEPA RSL	270	5.0	1.0	0.38
Bromoform	75-25-2	62,000	HH – USEPA RSL	62,000	5.0	2.5	1.1
Bromomethane	74-83-9	730	HH – USEPA RSL	730	5.0	1.0	0.58
Carbon Disulfide	75-15-0	82,000	HH – USEPA RSL	82,000	5.0	0.25	0.14
Carbon Tetrachloride	56-23-5	610	HH – USEPA RSL	610	5.0	1.0	0.45

SAP Worksheet #15.1: Reference Limits and Evaluation Tables (*continued*)

Matrix: Soil

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; pH

Analyte	CAS No.	PAL (µg/kg)	PAL Reference	Quantitation Goal (µg/kg)	Laboratory-Specific Limits (µg/kg)		
					LOQs	LODs	DLs
Chlorobenzene	108-90-7	29,000	HH – USEPA RSL	29,000	5.0	0.25	0.25
Chloroethane	75-00-3	1,500,000	HH – USEPA RSL	1,500,000	5.0	2.5	0.71
Chloroform	67-66-3	290	HH – USEPA RSL	290	5.0	1.0	0.31
Chloromethane	74-87-3	12,000	HH – USEPA RSL	12,000	5.0	1.0	0.32
Chloroprene	126-99-8	9.40	HH – USEPA RSL	9.40	5.0	1.0	0.39
cis-1,3-Dichloropropene	10061-01-5	100	Eco – CCME 2007	100	5.0	1.0	0.39
Dibromochloromethane	124-48-1	680	HH – USEPA RSL	680	5.0	1.0	0.56
Dibromomethane	74-95-3	2,500	HH – USEPA RSL	2,500	5.0	2.5	1.2
Dichlorodifluoromethane	75-71-8	9,400	HH – USEPA RSL	9,400	5.0	1.0	0.44
Ethyl benzene	100-41-4	5,003	Eco – MHSPE 2000	5,003	5.0	2.5	0.58
Ethyl methacrylate	97-63-2	150,000	HH – USEPA RSL	150,000	50	10	4.5
Iodomethane	74-88-4	NE	NA	NE	5.0	1.0	0.32
Isobutanol	78-83-1	1,800,000	HH – USEPA RSL	1,800,000	250	50	16
Methacrylonitrile	126-98-7	320	HH – USEPA RSL	320	50	2.5	2.2
Methyl methacrylate	80-62-6	480,000	HH – USEPA RSL	480,000	50	10	3.2
Methylene Chloride	75-09-2	1,040	Eco – MHSPE 2000	1,040	5.0	1.0	0.49
Pentachloroethane	76-01-7	5,400	HH – USEPA RSL	5,400	5.0	1.0	0.56
Propionitrile	107-12-0	NE	NA	NE	250	13	10
Styrene	100-42-5	10,030	Eco – MHSPE 2000	10,030	5.0	0.25	0.080
Tetrachloroethene	127-18-4	400	Eco – MHSPE 2000	400	5.0	2.5	1.6
Toluene	108-88-3	13,001	Eco – MHSPE 2000	13,001	5.0	10	0.34
trans-1,2-dichloroethene	156-60-5	100	Eco – CCME 2007	100	5.0	2.5	1.0
trans-1,3-Dichloropropene	10061-02-6	100	Eco – CCME 2007	100	5.0	1.0	0.41

SAP Worksheet #15.1: Reference Limits and Evaluation Tables (*continued*)

Matrix: Soil

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; pH

Analyte	CAS No.	PAL (µg/kg)	PAL Reference	Quantitation Goal (µg/kg)	Laboratory-Specific Limits (µg/kg)		
					LOQs	LODs	DLs
trans-1,4-Dichloro-2-butene	110-57-6	6.90	HH – USEPA RSL	6.90	**20**	4.0	1.8
Trichloroethene	79-01-6	910	HH – USEPA RSL	910	5.0	1.0	0.32
Trichlorofluoromethane	75-69-4	79,000	HH – USEPA RSL	79,000	5.0	1.0	0.30
Vinyl Acetate	108-05-4	97,000	HH – USEPA RSL	97,000	5.0	0.25	0.21
Vinyl Chloride	75-01-4	11.0	Eco – MHSPE 2000	11.0	5.0	1.0	0.42
Xylenes	108-38-3	1,000	Eco – USEPA 2003	1,000	5.0	0.50	0.14
Appendix IX LLPAHS (8270D-SIM)							
2-Methylnaphthalene	91-57-6	23,000	HH – USEPA RSL	23,000	8.3	2.0	0.91
Acenaphthene	83-32-9	340,000	HH – USEPA RSL	340,000	8.3	2.0	0.65
Acenaphthylene	208-96-8	340,000	HH – USEPA RSL	340,000	8.3	2.0	0.76
Anthracene	120-12-7	1,700,000	HH – USEPA RSL	1,700,000	8.3	2.0	0.56
Benzo(a)anthracene	56-55-3	150	HH – USEPA RSL	150	8.3	4.0	1.8
Benzo(a)pyrene	50-32-8	15.0	HH – USEPA RSL	15.0	8.3	4.0	1.7
Benzo(b)fluoranthene	205-99-2	150	HH – USEPA RSL	150	8.3	4.0	1.8
Benzo(g,h,i)perylene	191-24-2	170,000	HH – USEPA RSL	170,000	8.3	4.0	1.5
Benzo(k)fluoranthene	207-08-9	1,500	HH – USEPA RSL	1,500	8.3	4.0	1.6
Chrysene	218-01-9	15,000	HH – USEPA RSL	15,000	8.3	4.0	1.8
Dibenzo(a,h)anthracene	53-70-3	15.0	HH – USEPA RSL	15.0	8.3	4.0	1.6
Fluoranthene	206-44-0	230,000	HH – USEPA RSL	230,000	8.3	4.0	1.1
Fluorene	86-73-7	230,000	HH – USEPA RSL	230,000	8.3	2.0	0.63
Indeno(1,2,3-cd)pyrene	193-39-5	150	HH – USEPA RSL	150	8.3	4.0	1.9
Naphthalene	91-20-3	3,600	HH – USEPA RSL	3,600	8.3	2.0	0.90
Phenanthrene	85-01-8	170,000	HH – USEPA RSL	170,000	8.3	2.0	0.78
Pyrene	129-00-0	170,000	HH – USEPA RSL	170,000	8.3	4.0	1.9

SAP Worksheet #15.1: Reference Limits and Evaluation Tables (*continued*)

Matrix: Soil

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; pH

Analyte	CAS No.	PAL (µg/kg)	PAL Reference	Quantitation Goal (µg/kg)	Laboratory-Specific Limits (µg/kg)		
					LOQs	LODs	DLs
Appendix IX Pesticides (8081B)							
4,4'-DDD	72-54-8	93.0	Eco – USEPA 2007a	93.0	2.1	0.16	0.081
4,4'-DDE	72-55-9	93.0	Eco – USEPA 2007a	93.0	2.1	0.16	0.072
4,4'-DDT	50-29-3	93.0	Eco – USEPA 2007a	93.0	2.1	0.40	0.25
Aldrin	309-00-2	29.0	HH – USEPA RSL	29.0	0.83	0.20	0.067
Alpha-BHC	319-84-6	77.0	HH – USEPA RSL	77.0	0.83	0.20	0.088
beta-BHC	319-85-7	3.98	Eco – USEPA 2003	3.98	0.83	0.20	0.14
Chlordane	12789-03-6	224	Eco – USEPA 2003	224	0.83	0.20	0.081
Chlorobenzilate	510-15-6	4,400	HH – USEPA RSL	4,400	170	68	27
delta-BHC	319-86-8	201	Eco – MHSPE 2000	201	0.86	0.20	0.079
Dieldrin	60-57-1	0.022	Eco – USEPA 2007b	0.022	**2.1**	**0.16**	**0.056**
Endosulfan I	959-98-8	100	Eco – Friday 1998	100	0.83	0.20	0.077
Endosulfan II	33213-65-9	100	Eco – Friday 1998	100	2.1	0.16	0.089
Endosulfan sulfate	1031-07-8	100	Eco – Friday 1998	100	2.1	0.16	0.062
Endrin	72-20-8	401	Eco – MHSPE 2000	401	2.1	0.16	0.047
Endrin Aldehyde	7421-93-4	100	Eco – Friday 1998	100	2.1	0.40	0.13
gamma-BHC	58-89-9	5.00	Eco – USEPA 2003	5.00	0.83	0.063	0.040
Heptachlor	76-44-8	110	HH – USEPA RSL	110	0.83	0.20	0.12
Heptachlor epoxide	1024-57-3	53.0	HH – USEPA RSL	53.0	0.83	0.20	0.053
Isodrin	465-73-6	3.32	Eco – USEPA 2003	3.32	**170**	**68**	**32**
Kepone	143-50-0	49.0	HH – USEPA RSL	49.0	**170**	**170**	**170**
Methyoxychlor	72-43-5	100	Eco – Friday 1998	100	8.3	3.3	0.29
Toxaphene	8001-35-2	100	Eco – Friday 1998	100	83	33	15

SAP Worksheet #15.1: Reference Limits and Evaluation Tables (*continued*)

Matrix: Soil

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; pH

Analyte	CAS No.	PAL (mg/kg)	PAL Reference	Quantitation Goal (mg/kg)	Laboratory-Specific Limits (mg/kg)		
					LOQs	LODs	DLs
Appendix IX Metals (6020A/6010C/7471B)							
Antimony	7440-36-0	3.10	HH – USEPA RSL	3.10	1.00	0.500	0.115
Arsenic	7440-38-2	0.39	HH – USEPA RSL	0.39	**1.00**	0.250	0.169
Barium	7440-39-3	330	Eco – USEPA 2005c	330	5.00	2.50	0.110
Beryllium	7440-41-7	16.0	HH – USEPA RSL	16.0	0.500	0.250	0.0405
Cadmium	7440-43-9	0.77	Eco – USEPA 2005e	0.77	0.500	0.250	0.0332
Chromium	7440-47-3	26.0	Eco – USEPA 2008	26.0	6.00	0.500	0.149
Cobalt	7440-48-4	2.30	HH – USEPA RSL	2.30	0.500	0.250	0.0198
Copper	7440-50-8	28.0	Eco – USEPA 2007c	28.0	1.00	0.500	0.0265
Lead	7439-92-1	11.0	Eco – USEPA 2005g	11.0	0.500	0.250	0.0127
Mercury (7471B)	7439-97-6	0.10	Eco – Efroymson et al 1997a	0.10	0.0330	0.0150	0.00700
Nickel	7440-02-0	38.0	Eco – USEPA 2007d	38.0	0.500	0.250	0.0240
Selenium	7782-49-2	0.52	Eco – USEPA – 2007e	0.52	**2.50**	**1.25**	0.131
Silver	7440-22-4	4.20	Eco – USEPA 2006	4.20	0.500	0.250	0.0418
Thallium	7440-28-0	0.078	HH – USEPA RSL	0.078	**0.500**	**0.250**	0.0164
Tin (6010C)	7440-31-5	50.0	Eco – Efroymson et al. 1997b	50.0	10.0	5.00	0.612
Vanadium	7440-62-2	7.80	Eco – USEPA 2005h	7.80	2.50	1.25	0.260
Zinc	7440-66-6	46.0	Eco – USEPA 2007f	46.0	2.50	1.25	0.627
Total Petroleum Hydrocarbons (8015C)							
TPH DRO	9999-99-5	100	PREQB 2002	100	10	1.9	0.77
TPH GRO	9999-99-7	100	PREQB 2002	100	0.50	0.37	0.10
Miscellaneous Analytes							
pH (S.U.) (9045D)	NA	NA	NA	NA	NA	NA	NA

Notes:
The PALs represent the most stringent ecological or human health, risk-based screening values. A complete set of ecological and human health, risk-based screening values and associated references are provided in Appendix A.
Human health RSLs represent values for residential soil; noncarcinogenic RSLs based on a target hazard quotient of 0.1 for conservative screening purposes (see Appendix A).
Bold values exceed the PAL and quantitation goal.
CCME – Canadian Council of Ministers of the Environment
MHSPE – Ministry of Housing, Spatial Planning, and Environment
RSL – Regional Screening Level

SAP Worksheet #15.2: Reference Limits and Evaluation Tables

Matrix: Groundwater

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; TDS and TSS

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Appendix IX VOCs (8260B)							
1,1,1,2-Tetrachloroethane	630-20-6	0.50	HH – USEPA RSL	0.50	0.50	0.080	0.020
1,1,1-Trichloroethane	71-55-6	200	HH – USEPA MCL	200	0.50	0.080	0.020
1,1,2-Trichloroethane	79-00-5	0.24	HH – USEPA RSL	0.24	**0.50**	0.080	0.040
1,1-Dichloroethane	75-34-3	2.40	HH – USEPA RSL	2.40	0.50	0.080	0.020
1,1-Dichloroethene	75-35-4	7.00	HH – USEPA MCL	7.00	0.50	0.080	0.040
1,2,3-Trichloropropane	96-18-4	0.00065	HH – USEPA RSL	0.00065	**0.50**	**0.30**	**0.090**
1,2-Dibromo-3-chloropropane	96-12-8	0.00032	HH – USEPA RSL	0.00032	**0.50**	**0.30**	**0.25**
1,2-Dibromoethane	106-93-4	0.0065	HH – USEPA RSL	0.0065	**0.50**	**0.080**	**0.040**
1,2-Dichloroethane	107-06-02	0.15	HH – USEPA RSL	0.15	**0.50**	0.080	0.030
1,2-Dichloropropane	78-87-5	0.38	HH – USEPA RSL	0.38	**0.50**	0.30	0.12
2-Butanone	78-93-3	490	HH – USEPA RSL	490	2.5	0.40	0.28
2-Hexanone	591-78-6	3.40	HH – USEPA RSL	3.40	2.5	1.5	0.51
3-Chloro-1-propene	107-05-1	0.63	HH – USEPA RSL	0.63	0.50	0.080	0.050
4-Methyl-2-pentanone	108-10-1	100	HH – USEPA RSL	100	2.5	1.5	0.42
Acetone	67-64-1	1,000	Eco – USEPA 2007	1,000	2.5	1.5	0.44
Acetonitrile	75-05-8	13.0	HH – USEPA RSL	13.0	0.50	0.080	0.030
Acrolein	107-02-8	0.0041	HH – USEPA RSL	0.0041	**5.0**	**3.0**	**1.0**
Acrylonitrile	107-13-1	0.045	HH – USEPA RSL	0.045	**5.0**	**0.80**	**0.36**
Benzene	71-43-2	0.39	HH – USEPA RSL	0.39	**0.50**	0.080	0.030
Bromodichloromethane	75-27-4	0.12	HH – USEPA RSL	0.12	**0.50**	0.080	0.030
Bromoform	75-25-2	7.90	HH – USEPA RSL	7.90	0.50	0.080	0.030
Bromomethane	74-83-9	0.70	HH – USEPA RSL	0.70	0.50	0.30	0.11
Carbon Disulfide	75-15-0	15.0	Eco – USEPA 2003	15.0	0.50	0.080	0.020
Carbon Tetrachloride	56-23-5	0.39	HH – USEPA RSL	0.39	**0.50**	0.080	0.030

SAP Worksheet #15.2: Reference Limits and Evaluation Tables (*continued*)

Matrix: Groundwater

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; TDS and TSS

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Chlorobenzene	108-90-7	7.20	HH – USEPA RSL	7.20	0.50	0.080	0.020
Chloroethane	75-00-3	2,100	HH – USEPA RSL	2,100	0.50	0.30	0.20
Chloroform	67-66-3	0.19	HH – USEPA RSL	0.19	**0.50**	0.080	0.030
Chloromethane	74-87-3	19.0	HH – USEPA RSL	19.0	0.50	0.30	0.11
Chloroprene	126-99-8	0.016	HH – USEPA RSL	0.016	**0.50**	**0.080**	**0.020**
cis-1,3-Dichloropropene	10061-01-5	0.41	HH – USEPA RSL	0.41	**0.50**	0.080	0.040
Dibromochloromethane	124-48-1	0.15	HH – USEPA RSL	0.15	**0.50**	0.080	0.040
Dibromomethane	75-95-3	0.79	HH – USEPA RSL	0.79	0.50	0.080	0.030
Dichlorodifluoromethane	75-71-8	19.0	HH – USEPA RSL	19.0	0.50	0.080	0.030
Ethyl benzene	100-41-4	1.30	HH – USEPA RSL	1.30	0.50	0.080	0.040
Ethyl methacrylate	97-63-5	42.0	HH – USEPA RSL	42.0	5.0	0.80	0.41
Iodomethane	74-88-4	NE	NA	NE	0.50	0.080	0.030
Isobutanol	78-83-1	460	HH – USEPA RSL	460	25	15	5.9
Methacrylonitrile	126-98-7	0.075	HH – USEPA RSL	0.075	**5.0**	**0.80**	**0.39**
Methyl methacrylate	80-62-6	140	HH – USEPA RSL	140	5.0	0.80	0.56
Methylene Chloride	75-09-2	5.0	HH – USEPA MCL	5.0	0.50	0.080	0.020
Pentachloroethane	76-01-7	0.56	HH – USEPA RSL	0.56	0.50	0.080	0.070
Propionitrile	107-12-0	15,200	Eco – USEPA 2007	15,200	25	4.0	1.4
Styrene	700-42-5	100	HH – USEPA MCL	100	0.50	0.080	0.020
Tetrachloroethene	127-18-4	5.0	HH – USEPA MCL	5.0	**0.50**	0.080	0.060
Toluene	108-88-3	37.0	Eco – USEPA 2001	37.0	0.50	0.080	0.030
trans-1,2-dichloroethene	156-60-5	8.60	HH – USEPA RSL	8.60	0.50	0.080	0.050
trans-1,3-Dichloropropene	10061-02-6	0.41	HH – USEPA RSL	0.41	**0.50**	0.080	0.050
trans-1,4-Dichloro-2-butene	110-57-6	0.0012	HH – USEPA RSL	0.0012	**2.0**	**1.2**	**0.33**
Trichloroethene	79-01-6	0.44	HH – USEPA RSL	0.44	**0.50**	0.080	0.030

SAP Worksheet #15.2: Reference Limits and Evaluation Tables (*continued*)

Matrix: Groundwater

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; TDS and TSS

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Trichlorofluoromethane	75-69-4	110	HH – USEPA RSL	110	0.50	0.080	0.060
Vinyl Acetate	108-05-4	41.0	HH – USEPA RSL	41.0	1.0	0.16	0.060
Vinyl Chloride	75-01-4	0.015	HH – USEPA RSL	0.015	**0.50**	**0.080**	**0.070**
Xylenes	108-38-3	19.0	HH – USEPA RSL	19.0	0.50	0.080	0.030
Appendix IX LLPAHs (8270D-SIM)							
2-Methylnaphthalene	91-57-6	2.70	HH – USEPA RSL	2.70	0.200	0.0500	0.0260
Acenaphthene	83-32-9	9.70	Eco – USEPA 2001	9.70	0.200	0.0500	0.0260
Acenaphthylene	208-96-8	6.00	Eco – Buchman 2008	6.00	0.200	0.0500	0.0220
Anthracene	120-12-7	5.35	Eco – USEPA 2007	5.35	0.200	0.0500	0.0180
Benzo(a)anthracene	56-55-3	0.025	Eco – USEPA 2003	0.025	**0.200**	**0.0500**	0.0160
Benzo(a)pyrene	50-32-8	0.0029	HH – USEPA RSL	0.0029	**0.200**	**0.0500**	**0.0150**
Benzo(b)fluoranthene	205-99-2	0.029	HH – USEPA RSL	0.029	**0.200**	**0.0500**	0.0130
Benzo(g,h,i)perylene	191-24-2	6.00	Eco – Buchman 2008	6.00	0.200	0.0500	0.0400
Benzo(k)fluoranthene	207-08-9	0.038	PRWQS	0.038	**0.200**	**0.0500**	0.0210
Chrysene	215-01-9	0.038	PRWQS	0.038	**0.200**	**0.0500**	0.0150
Dibenzo(a,h)anthracene	53-70-3	0.0029	HH – USEPA RSL	0.0029	**0.200**	**0.0500**	**0.0290**
Fluoranthene	206-44-0	11.0	Eco – USEPA 1996	11.0	0.200	0.0500	0.0210
Fluorene	86-73-7	10.0	Eco – USEPA 2007	10.0	0.200	0.0500	0.0230
Indeno(1,2,3-cd)pyrene	193-39-5	0.029	HH – USEPA RSL	0.029	**0.200**	**0.0500**	**0.0320**
Naphthalene	91-20-3	0.14	HH – USEPA RSL	0.14	**0.200**	0.0500	0.0160
Phenanthrene	85-01-8	8.30	Eco – USEPA 1996	8.30	0.200	0.0500	0.0170
Pyrene	129-00-0	0.248	Eco – USEPA 2007	0.248	0.200	0.0500	0.0420
Appendix IX Pesticides (8081B)							
4,4'-DDD	72-54-8	0.001	Eco – PREQB 2010	0.001	**0.050**	**0.011**	**0.0055**
4,4'-DDE	72-55-9	0.001	Eco – PREQB 2010	0.001	**0.050**	**0.011**	**0.0039**

SAP Worksheet #15.2: Reference Limits and Evaluation Tables (*continued*)

Matrix: Groundwater

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; TDS and TSS

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
4,4'-DDT	50-29-3	0.001	Eco – PREQB 2010	0.001	**0.050**	**0.011**	**0.0051**
Aldrin	309-00-2	0.00021	HH – USEPA RSL	0.00021	**0.025**	**0.0045**	**0.0027**
Alpha-BHC	319-84-6	0.0062	HH – USEPA RSL	0.0062	**0.025**	0.0045	0.0019
beta-BHC	319-85-7	0.022	HH – USEPA RSL	0.022	**0.025**	0.010	0.0095
Chlordane	12789-03-6	0.027	HH – USEPA RSL	0.19	0.027	0.0045	0.0020
Chlorobenzilate	510-15-6	0.076	Eco – USEPA 2005	0.076	**5.0**	**2.0**	**1.5**
delta-BHC	319-86-8	0.022	HH – USEPA RSL	0.022	**0.025**	0.0045	0.0032
Dieldrin	60-57-1	0.00052	PRWQS	0.00052	**0.050**	**0.011**	**0.0051**
Endosulfan I	959-98-8	0.0087	Eco – PREQB 2010	0.0087	**0.025**	0.0045	0.0043
Endosulfan II	33213-65-9	0.0087	Eco – PREQB 2010	0.0087	**0.050**	**0.011**	0.0055
Endosulfan sulfate	1031-07-8	0.92	Eco – USEPA 2007	0.92	0.050	0.011	0.0068
Endrin	72-20-8	0.0023	Eco – PREQB 2010	0.0023	**0.050**	**0.011**	**0.0069**
Endrin Aldehyde	7421-93-4	0.17	HH – USEPA RSL	0.17	0.050	0.019	0.012
gamma-BHC	58-89-9	0.036	HH – USEPA RSL	0.036	0.025	0.0045	0.0019
Heptachlor	76-44-8	0.00079	PRWQS	0.00079	**0.025**	**0.0045**	**0.0024**
Heptachlor epoxide	1024-57-3	0.0033	HH – USEPA RSL	0.0033	**0.025**	**0.0045**	0.0028
Isodrin	465-73-6	0.12	Eco – USEPA 2007	0.12	**5.0**	**2.0**	**0.65**
Kepone	143-50-0	0.003	HH – USEPA RSL	0.003	**5.0**	**5.0**	**5.0**
Methyoxychlor	72-43-5	0.03	Eco – PREQB 2010	0.03	**0.25**	0.025	0.015
Toxaphene	8001-35-2	0.0002	Eco – PREQB 2010	0.0002	**2.5**	**1.0**	**0.48**
Appendix IX Metals (6020A/6010C/7470A)							
Antimony	7440-36-0	0.60	HH – USEPA RSL	0.60	**2.00**	**1.00**	0.130
Arsenic	7440-38-2	0.045	HH – USEPA RSL	0.045	**1.00**	**0.500**	**0.110**
Barium	7440-39-3	290	HH – USEPA RSL	290	10.0	5.00	0.130
Beryllium	7440-41-7	1.60	HH – USEPA RSL	1.60	1.00	0.500	0.150

SAP Worksheet #15.2: Reference Limits and Evaluation Tables (*continued*)

Matrix: Groundwater

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; TDS and TSS

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Cadmium	7440-43-9	0.69	HH – USEPA RSL	0.69	**1.00**	0.500	0.140
Chromium	7440-47-3	50.4	Eco – PREQB 2010	50.4	2.00	1.00	0.140
Cobalt	7440-48-4	0.47	HH – USEPA RSL	0.47	**1.00**	**0.500**	0.0400
Copper	7440-50-8	3.73	Eco – PREQB 2010	3.73	2.00	1.00	0.0620
Lead	7439-92-1	8.52	Eco – PREQB 2010	8.52	1.00	0.500	0.0600
Mercury (7470A)	7439-97-6	0.05	PRWQS	0.05	**0.200**	**0.150**	0.0355
Nickel	7440-02-0	8.28	Eco – PREQB 2010	8.28	1.00	0.500	0.0980
Selenium	7782-49-2	7.80	HH – USEPA RSL	7.80	5.00	2.50	0.380
Silver	7440-22-4	2.24	Eco – PREQB 2010	2.24	1.00	0.500	0.0400
Thallium	7440-28-0	0.016	HH – USEPA RSL	0.016	**1.00**	**0.500**	**0.0430**
Tin (6010C)	7440-31-5	180	Eco – USEPA 2003	180	50.0	25.0	2.87
Vanadium	7440-62-2	7.80	HH – USEPA RSL	7.80	5.00	2.50	0.160
Zinc	7440-66-6	85.6	Eco – PREQB 2010	85.6	2.00	1.00	2.00
Total Petroleum Hydrocarbons (8015C)							
TPH DRO	9999-99-5	50,000	PREQB 2002	50,000	500	150	91
TPH GRO	9999-99-7	50,000	PREQB 2002	50,000	500	420	170
Miscellaneous Analytes (mg/L)							
TDS (2540C)	NA	NE	NA	NE	10.0	6.00	3.00
TSS (2540D)	NA	NE	NA	NE	4.00	4.00	2.00

Notes:
The PALs represent the most stringent ecological or human health, risk-based screening values. A complete set of ecological and human health, risk-based screening values and associated references are provided in Appendix A.
Human health RSLs represent values for tapwater; noncarcinogenic RSLs based on a target hazard quotient of 0.1 for conservative screening purposes (see Appendix A).
Bold values exceed the PAL and quantitation goal.
MCL – Maximum Contaminant Level
PRWQS – Puerto Rico Water Quality Standard

SAP Worksheet #15.3: Reference Limits and Evaluation Tables

Matrix: Sediment

Analytical Group: Appendix IX LLPAHs, Pesticides, and Metals; TOC

Analyte	CAS No.	PAL (µg/kg)	PAL Reference	Quantitation Goal (µg/kg)	Laboratory-Specific Limits (µg/kg)		
					LOQs	LODs	DLs
Appendix IX LLPAHs (8270D-SIM)							
2-Methylnaphthalene	91-57-6	20.2	Eco – MacDonald 1994	20.2	8.3	2.0	0.91
Acenaphthene	83-32-9	6.71	Eco – MacDonald 1994	6.71	**8.3**	2.0	0.65
Acenaphthylene	208-96-8	5.87	Eco – MacDonald 1994	5.87	**8.3**	2.0	0.76
Anthracene	120-12-7	46.9	Eco – MacDonald 1994	46.9	8.3	2.0	0.56
Benzo(a)anthracene	56-55-3	74.8	Eco – MacDonald 1994	74.8	8.3	4.0	1.8
Benzo(a)pyrene	50-32-8	15.0	HH – USEPA RSL	15.0	8.3	4.0	1.7
Benzo(b)fluoranthene	205-99-2	150	HH – USEPA RSL	150	8.3	4.0	1.8
Benzo(g,h,i)perylene	191-24-2	670	Eco – Buchman 2008	670	8.3	4.0	1.5
Benzo(k)fluoranthene	207-08-9	1,500	HH – USEPA RSL	1,500	8.3	4.0	1.6
Chrysene	218-01-9	108	Eco – MacDonald 1994	108	8.3	4.0	1.8
Dibenzo(a,h)anthracene	53-70-3	6.22	Eco – MacDonald 1994	6.22	**8.3**	4.0	1.6
Fluoranthene	206-44-0	113	Eco – MacDonald 1994	113	8.3	4.0	1.1
Fluorene	86-73-7	21.2	Eco – MacDonald 1994	21.2	8.3	2.0	0.63
Indeno(1,2,3-cd)pyrene	193-39-5	150	HH – USEPA RSL	150	8.3	4.0	1.9
Naphthalene	91-20-3	34.6	Eco – MacDonald 1994	34.6	8.3	2.0	0.90
Phenanthrene	85-01-8	86.7	Eco – MacDonald 1994	86.7	8.3	2.0	0.78
Pyrene	129-00-0	153	Eco – MacDonald 1994	153	8.3	4.0	1.9
Appendix IX Pesticides (8081B)							
4,4'-DDD	72-54-8	1.22	Eco – MacDonald 1994	1.22	**2.1**	0.16	0.081
4,4'-DDE	72-55-9	2.07	Eco – MacDonald 1994	2.07	**2.1**	0.16	0.072
4,4'-DDT	50-29-3	1.19	Eco – MacDonald 1994	1.19	**2.1**	0.40	0.25
Aldrin	309-00-2	9.50	Eco – Buchman 2008	9.50	0.83	0.20	0.067
Alpha-BHC	319-84-6	6.00	Eco – Persaud et al. 1993	6.00	0.83	0.20	0.088

SAP Worksheet #15.3: Reference Limits and Evaluation Tables (*continued*)

Matrix: Sediment

Analytical Group: Appendix IX LLPAHs, Pesticides, and Metals; TOC

Analyte	CAS No.	PAL (µg/kg)	PAL Reference	Quantitation Goal (µg/kg)	Laboratory-Specific Limits (µg/kg)		
					LOQs	LODs	DLs
beta-BHC	319-85-7	5.00	Eco – Persaud et al. 1993	5.00	0.83	0.20	0.14
Chlordane	12789-03-6	2.26	Eco – MacDonald et al. 2000	2.26	0.83	0.20	0.081
Chlorobenzilate	510-15-6	34.7	Eco – USEPA 1993 and 1996	34.7	**170**	**68**	27
delta-BHC	319-86-8	6.96	Eco – USEPA 1993 and 1996	6.96	0.86	0.20	0.079
Dieldrin	60-57-1	0.715	Eco – MacDonald 1994	0.715	**2.1**	0.16	0.056
Endosulfan I	959-98-8	0.51	Eco – USEPA 1993 and 1996	0.51	**0.83**	0.20	0.077
Endosulfan II	33213-65-9	0.51	Eco – USEPA 1993 and 1996	0.51	**2.1**	0.16	0.089
Endosulfan sulfate	1031-07-8	36.5	Eco – USEPA 1993 and 1996	36.5	2.1	0.16	0.062
Endrin	72-20-8	2.22	Eco – MacDonald et al. 2000	2.22	2.1	0.16	0.047
Endrin Aldehyde	7421-93-4	2.22	Value for Endrin used as surrogate	2.22	2.1	0.40	0.13
gamma-BHC	58-89-9	0.32	Eco – MacDonald 1994	0.32	**0.83**	0.063	0.040
Heptachlor	76-44-8	0.30	Eco – Buchman 2008	0.30	**0.83**	0.20	0.12
Heptachlor epoxide	1024-57-3	2.47	Eco – MacDonald et al. 2000	2.47	0.83	0.20	0.053
Isodrin	465-73-6	2,944	Eco – USEPA 1993 and 1996	2,944	170	68	32
Kepone	143-50-0	49.0	HH – USEPA RSL	49.0	**170**	**170**	**170**
Methyoxychlor	72-43-5	29.6	Eco – USEPA 1993 and 1996	29.6	8.3	3.3	0.29
Toxaphene	8001-35-2	0.10	Eco – MacDonald et al. 2000	0.10	**83**	**33**	**15**

SAP Worksheet #15.3: Reference Limits and Evaluation Tables (*continued*)

Matrix: Sediment

Analytical Group: Appendix IX LLPAHs, Pesticides, and Metals; TOC

Analyte	CAS No.	PAL (mg/kg)	PAL Reference	Quantitation Goal (mg/kg)	Laboratory-Specific Limits (mg/kg)		
					LOQs	LODs	DLs
Appendix IX Metals (6020A/6010C/7471B)							
Antimony	7440-36-0	2.00	Eco – Long and Morgan 1991	2.00	1.00	0.500	0.115
Arsenic	7440-38-2	0.39	HH – USEPA RSL	0.39	**1.00**	0.250	0.169
Barium	7440-39-3	48	Eco – Buchman 2008	48	5.00	2.50	0.110
Beryllium	7440-41-7	16.0	HH – USEPA RSL	16.0	0.500	0.250	0.0405
Cadmium	7440-43-9	0.676	Eco – MacDonald 1994	0.676	0.500	0.250	0.0332
Chromium	7440-47-3	52.3	Eco – MacDonald 1994	52.3	6.00	0.500	0.149
Cobalt	7440-48-4	2.30	HH – USEPA RSL	2.30	0.500	0.250	0.0198
Copper	7440-50-8	18.7	Eco – MacDonald 1994	18.7	1.00	0.500	0.0265
Lead	7439-92-1	30.2	Eco – MacDonald 1994	30.2	0.500	0.250	0.0127
Mercury (7471B)	7439-97-6	0.13	Eco – MacDonald 1994	0.13	0.0330	0.0150	0.00700
Nickel	7440-02-0	15.9	Eco – MacDonald 1994	15.9	0.500	0.250	0.0240
Selenium	7782-49-2	1.00	Eco – Buchman 2008	1.00	**2.50**	**1.25**	0.131
Silver	7440-22-4	0.733	Eco – MacDonald 1994	0.733	0.500	0.250	0.0418
Thallium	7440-28-0	0.78	HH – USEPA RSL	0.78	0.500	0.250	0.0164
Tin (6010C)	7440-31-5	3.40	Eco – Buchman 2008	3.40	**10.0**	**5.00**	0.612
Vanadium	7440-62-2	39.0	HH – USEPA RSL	39.0	2.50	1.25	0.260
Zinc	7440-66-6	124	Eco – MacDonald 1994	124	2.50	1.25	0.627
Miscellaneous Analytes							
TOC (Lloyd Kahn)	--	NE	NA	NE	100.0	75.00	33.01

Notes:
The PALs represent the most stringent ecological or human health, risk-based screening values. A complete set of ecological and human health, risk-based screening values and associated references are provided in Appendix A.
Human health RSLs represent values for residential soil; noncarcinogenic RSLs based on a target hazard quotient of 0.1 for conservative screening purposes (see Appendix A).
Bold values exceed the PAL and quantitation goal.

SAP Worksheet #15.4: Reference Limits and Evaluation Tables

Matrix: Solid IDW

Analytical Group: TCLP VOCs, Pesticides, and Metals; Appendix IX LLPAHs; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL	PAL Reference	Quantitation Goal	Laboratory-Specific Limits		
					LOQs	LODs	DLs
TCLP VOCs (µg/L) (1311/8260B)							
1,1-Dichloroethene	75-35-4	700	40 CFR Part 261	700	5.0	2.0	0.53
1,2-Dichloroethane	107-06-2	500	40 CFR Part 261	500	5.0	0.50	0.23
2-Butanone	78-93-6	200,000	40 CFR Part 261	200,000	12	5.0	1.5
Benzene	71-43-2	500	40 CFR Part 261	500	5.0	0.50	0.28
Carbon Tetrachloride	56-23-5	500	40 CFR Part 261	500	5.0	0.50	0.31
Chlorobenzene	108-90-7	100,000	40 CFR Part 261	100,000	5.0	0.50	0.27
Chloroform	67-66-3	6,000	40 CFR Part 261	6,000	5.0	0.50	0.27
Tetrachloroethene	127-18-4	700	40 CFR Part 261	700	5.0	0.50	0.28
Trichloroethene	79-01-6	500	40 CFR Part 261	500	5.0	0.50	0.42
Vinyl Chloride	75-01-4	200	40 CFR Part 261	200	5.0	0.50	0.48
TCLP Pesticides (µg/L) (1311/8081B)							
Chlordane	12789-03-6	30	40 CFR Part 261	30	0.80	0.50	0.24
Endrin	72-20-8	20	40 CFR Part 261	20	0.050	0.011	0.0069
Gamma-BHC	58-89-9	400	40 CFR Part 261	400	0.025	0.0045	0.0019
Heptachlor	76-44-8	8	40 CFR Part 261	8	0.025	0.0045	0.0024
Heptachlor epoxide	1024-57-3	8	40 CFR Part 261	8	0.025	0.0045	0.0028
Methoxychlor	72-43-5	10,000	40 CFR Part 261	10,000	0.25	0.025	0.015
Toxaphene	8001-35-2	500	40 CFR Part 261	500	2.5	1.0	0.48
TCLP Metals (µg/L) (1311/6010C/7470A)							
Arsenic	7440-38-2	5,000	40 CFR Part 261	5,000	10	5.0	3.9
Barium	7440-39-3	100,000	40 CFR Part 261	100,000	200	100	66
Cadmium	7440-43-9	1,000	40 CFR Part 261	1,000	5.0	2.5	1.8
Chromium	7440-47-3	5,000	40 CFR Part 261	5,000	10	5.0	3.8
Lead	7439-92-1	5,000	40 CFR Part 261	5,000	10	5.0	3.6

SAP Worksheet #15.4: Reference Limits and Evaluation Tables (*continued*)

Matrix: Solid IDW

Analytical Group: TCLP VOCs, Pesticides, and Metals; Appendix IX LLPAHs; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL	PAL Reference	Quantitation Goal	Laboratory-Specific Limits		
					LOQs	LODs	DLs
Mercury (7470A)	7439-97-6	200	40 CFR Part 261	200	0.200	0.150	0.0355
Selenium	7782-49-2	1,000	40 CFR Part 261	1,000	10	5.0	3.5
Silver	7440-22-4	5,000	40 CFR Part 261	5,000	5.0	2.5	2.0
Appendix IX LLPAHs (µg/L) (8270D-SIM)							
2-Methylnaphthalene	91-57-6	NA	NA	8.3	8.3	2.0	0.91
Acenaphthene	83-32-9	NA	NA	8.3	8.3	2.0	0.65
Acenaphthylene	208-96-8	NA	NA	8.3	8.3	2.0	0.76
Anthracene	120-12-7	NA	NA	8.3	8.3	2.0	0.56
Benzo(a)anthracene	56-55-3	NA	NA	8.3	8.3	4.0	1.8
Benzo(a)pyrene	50-32-8	NA	NA	8.3	8.3	4.0	1.7
Benzo(b)fluoranthene	205-99-2	NA	NA	8.3	8.3	4.0	1.8
Benzo(g,h,i)perylene	191-24-2	NA	NA	8.3	8.3	4.0	1.5
Benzo(k)fluoranthene	207-08-9	NA	NA	8.3	8.3	4.0	1.6
Chrysene	218-01-9	NA	NA	8.3	8.3	4.0	1.8
Dibenzo(a,h)anthracene	53-70-3	NA	NA	8.3	8.3	4.0	1.6
Fluoranthene	206-44-0	NA	NA	8.3	8.3	4.0	1.1
Fluorene	86-73-7	NA	NA	8.3	8.3	2.0	0.63
Indeno(1,2,3-cd)pyrene	193-39-5	NA	NA	8.3	8.3	4.0	1.9
Naphthalene	91-20-3	NA	NA	8.3	8.3	2.0	0.90
Phenanthrene	85-01-8	NA	NA	8.3	8.3	2.0	0.78
Pyrene	129-00-0	NA	NA	8.3	8.3	4.0	1.9
Total Petroleum Hydrocarbons (mg/kg) (8015C)							
TPH DRO	9999-99-5	NA	NA	10	10	1.9	0.77
TPH GRO	9999-99-7	NA	NA	0.50	0.50	0.37	0.10

SAP Worksheet #15.4: Reference Limits and Evaluation Tables (*continued*)

Matrix: Solid IDW

Analytical Group: TCLP VOCs, Pesticides, and Metals; Appendix IX LLPAHs; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL	PAL Reference	Quantitation Goal	Laboratory-Specific Limits		
					LOQs	LODs	DLs
RCRA Hazardous Waste Characteristics							
Flashpoint/Ignitability (^{o}F) (ASTM D93-80)	NA	NA	NA	NA	NA	NA	NA
Reactive Cyanide (mg/kg) (9014)	NA	NA	NA	125	125	NA	1.00
Reactive Sulfide (mg/kg) (9034)	NA	NA	NA	125	125	NA	1.00
pH (S.U.) (9040C)	NA	NA	NA	NA	NA	NA	NA

Note:
CFR – Code of Federal Regulations

SAP Worksheet #15.5: Reference Limits and Evaluation Tables

Matrix: Liquid IDW

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Appendix IX VOCs (8260B)							
1,1,1,2-Tetrachloroethane	630-20-6	NA	NA	0.50	0.50	0.080	0.020
1,1,1-Trichloroethane	71-55-6	NA	NA	0.50	0.50	0.080	0.020
1,1,2-Trichloroethane	79-00-5	NA	NA	0.50	0.50	0.080	0.040
1,1-Dichloroethane	75-34-3	NA	NA	0.50	0.50	0.080	0.020
1,1-Dichloroethene	75-35-4	NA	NA	0.50	0.50	0.080	0.040
1,2,3-Trichloropropane	96-18-4	NA	NA	0.50	0.50	0.30	0.090
1,2-Dibromo-3-chloropropane	96-12-8	NA	NA	0.50	0.50	0.30	0.25
1,2-Dibromoethane	106-93-4	NA	NA	0.50	0.50	0.080	0.040
1,2-Dichloroethane	107-06-02	NA	NA	0.50	0.50	0.080	0.030
1,2-Dichloropropane	78-87-5	NA	NA	0.50	0.50	0.30	0.12
2-Butanone	78-93-3	NA	NA	2.5	2.5	0.40	0.28
2-Hexanone	591-78-6	NA	NA	2.5	2.5	1.5	0.51
3-Chloro-1-propene	107-05-1	NA	NA	0.50	0.50	0.080	0.050
4-Methyl-2-pentanone	108-10-1	NA	NA	2.5	2.5	1.5	0.42
Acetone	67-64-1	NA	NA	2.5	2.5	1.5	0.44
Acetonitrile	75-05-8	NA	NA	0.50	0.50	0.080	0.030
Acrolein	107-02-8	NA	NA	5.0	5.0	3.0	1.0
Acrylonitrile	107-13-1	NA	NA	5.0	5.0	0.80	0.36
Benzene	71-43-2	NA	NA	0.50	0.50	0.080	0.030
Bromodichloromethane	75-27-4	NA	NA	0.50	0.50	0.080	0.030
Bromoform	75-25-2	NA	NA	0.50	0.50	0.080	0.030
Bromomethane	74-83-9	NA	NA	0.50	0.50	0.30	0.11
Carbon Disulfide	75-15-0	NA	NA	0.50	0.50	0.080	0.020
Carbon Tetrachloride	56-23-5	NA	NA	0.50	0.50	0.080	0.030

SAP Worksheet #15.5: Reference Limits and Evaluation Tables (*continued*)

Matrix: Liquid IDW

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Chlorobenzene	108-90-7	NA	NA	0.50	0.50	0.080	0.020
Chloroethane	75-00-3	NA	NA	0.50	0.50	0.30	0.20
Chloroform	67-66-3	NA	NA	0.50	0.50	0.080	0.030
Chloromethane	74-87-3	NA	NA	0.50	0.50	0.30	0.11
Chloroprene	126-99-8	NA	NA	0.50	0.50	0.080	0.020
cis-1,3-Dichloropropene	10061-01-5	NA	NA	0.50	0.50	0.080	0.040
Dibromochloromethane	124-48-1	NA	NA	0.50	0.50	0.080	0.040
Dibromomethane	75-95-3	NA	NA	0.50	0.50	0.080	0.030
Dichlorodifluoromethane	75-71-8	NA	NA	0.50	0.50	0.080	0.030
Ethyl benzene	100-41-4	NA	NA	0.50	0.50	0.080	0.040
Ethyl methacrylate	97-63-5	NA	NA	5.0	5.0	0.80	0.41
Iodomethane	74-88-4	NA	NA	0.50	0.50	0.080	0.030
Isobutanol	78-83-1	NA	NA	25	25	15	5.9
Methacrylonitrile	126-98-7	NA	NA	5.0	5.0	0.80	0.39
Methyl methacrylate	80-62-6	NA	NA	5.0	5.0	0.80	0.56
Methylene Chloride	75-09-2	NA	NA	0.50	0.50	0.080	0.020
Pentachloroethane	76-01-7	NA	NA	0.50	0.50	0.080	0.070
Propionitrile	107-12-0	NA	NA	25	25	4.0	1.4
Styrene	700-42-5	NA	NA	0.50	0.50	0.080	0.020
Tetrachloroethene	127-18-4	NA	NA	0.50	0.50	0.080	0.060
Toluene	108-88-3	NA	NA	0.50	0.50	0.080	0.030
trans-1,2-dichloroethene	156-60-5	NA	NA	0.50	0.50	0.080	0.050
trans-1,3-Dichloropropene	10061-02-6	NA	NA	0.50	0.50	0.080	0.050
trans-1,4-Dichloro-2-butene	110-57-6	NA	NA	2.0	2.0	1.2	0.33
Trichloroethene	79-01-6	NA	NA	0.50	0.50	0.080	0.030

SAP Worksheet #15.5: Reference Limits and Evaluation Tables (*continued*)

Matrix: Liquid IDW

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Trichlorofluoromethane	75-69-4	NA	NA	0.50	0.50	0.080	0.060
Vinyl Acetate	108-05-4	NA	NA	1.0	1.0	0.16	0.060
Vinyl Chloride	75-01-4	NA	NA	0.50	0.50	0.080	0.070
Xylenes	108-38-3	NA	NA	0.50	0.50	0.080	0.030
Appendix IX LLPAHs (8270D-SIM)							
2-Methylnaphthalene	91-57-6	NA	NA	0.200	0.200	0.0500	0.0260
Acenaphthene	83-32-9	NA	NA	0.200	0.200	0.0500	0.0260
Acenaphthylene	208-96-8	NA	NA	0.200	0.200	0.0500	0.0220
Anthracene	120-12-7	NA	NA	0.200	0.200	0.0500	0.0180
Benzo(a)anthracene	56-55-3	NA	NA	0.200	0.200	0.0500	0.0160
Benzo(a)pyrene	50-32-8	NA	NA	0.200	0.200	0.0500	0.0150
Benzo(b)fluoranthene	205-99-2	NA	NA	0.200	0.200	0.0500	0.0130
Benzo(g,h,i)perylene	191-24-2	NA	NA	0.200	0.200	0.0500	0.0400
Benzo(k)fluoranthene	207-08-9	NA	NA	0.200	0.200	0.0500	0.0210
Chrysene	215-01-9	NA	NA	0.200	0.200	0.0500	0.0150
Dibenzo(a,h)anthracene	53-70-3	NA	NA	0.200	0.200	0.0500	0.0290
Fluoranthene	206-44-0	NA	NA	0.200	0.200	0.0500	0.0210
Fluorene	86-73-7	NA	NA	0.200	0.200	0.0500	0.0230
Indeno(1,2,3-cd)pyrene	193-39-5	NA	NA	0.200	0.200	0.0500	0.0320
Naphthalene	91-20-3	NA	NA	0.200	0.200	0.0500	0.0160
Phenanthrene	85-01-8	NA	NA	0.200	0.200	0.0500	0.0170
Pyrene	129-00-0	NA	NA	0.200	0.200	0.0500	0.0420
Appendix IX Pesticides (8081B)							
4,4'-DDD	72-54-8	NA	NA	0.050	0.050	0.011	0.0055
4,4'-DDE	72-55-9	NA	NA	0.050	0.050	0.011	0.0039

SAP Worksheet #15.5: Reference Limits and Evaluation Tables (*continued*)

Matrix: Liquid IDW

Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
4,4'-DDT	50-29-3	NA	NA	0.050	0.050	0.011	0.0051
Aldrin	309-00-2	NA	NA	0.025	0.025	0.0045	0.0027
Alpha-BHC	319-84-6	NA	NA	0.025	0.025	0.0045	0.0019
beta-BHC	319-85-7	NA	NA	0.025	0.025	0.010	0.0095
Chlordane	12789-03-6	NA	NA	0.025	0.025	0.0045	0.0020
Chlorobenzilate	510-15-6	NA	NA	5.0	5.0	2.0	1.5
delta-BHC	319-86-8	NA	NA	0.025	0.025	0.0045	0.0032
Dieldrin	60-57-1	NA	NA	0.050	0.050	0.011	0.0051
Endosulfan I	959-98-8	NA	NA	0.025	0.025	0.0045	0.0043
Endosulfan II	33213-65-9	NA	NA	0.050	0.050	0.011	0.0055
Endosulfan sulfate	1031-07-8	NA	NA	0.050	0.050	0.011	0.0068
Endrin	72-20-8	NA	NA	0.050	0.050	0.011	0.0069
Endrin Aldehyde	7421-93-4	NA	NA	0.050	0.050	0.019	0.012
gamma-BHC	58-89-9	NA	NA	0.025	0.025	0.0045	0.0019
Heptachlor	76-44-8	NA	NA	0.025	0.025	0.0045	0.0024
Heptachlor epoxide	1024-57-3	NA	NA	0.025	0.025	0.0045	0.0028
Isodrin	465-73-6	NA	NA	5.0	5.0	2.0	0.65
Kepone	143-50-0	NA	NA	5.0	5.0	5.0	5.0
Methyoxychlor	72-43-5	NA	NA	0.25	0.25	0.025	0.015
Toxaphene	8001-35-2	NA	NA	2.5	2.5	1.0	0.48
Appendix IX Metals (6020A/6010C/7470A)							
Antimony	7440-36-0	NA	NA	2.00	2.00	1.00	0.130
Arsenic	7440-38-2	NA	NA	1.00	1.00	0.500	0.110
Barium	7440-39-3	NA	NA	10.0	10.0	5.00	0.130
Beryllium	7440-41-7	NA	NA	1.00	1.00	0.500	0.150

SAP Worksheet #15.5: Reference Limits and Evaluation Tables (*continued*)

Matrix: Liquid IDW

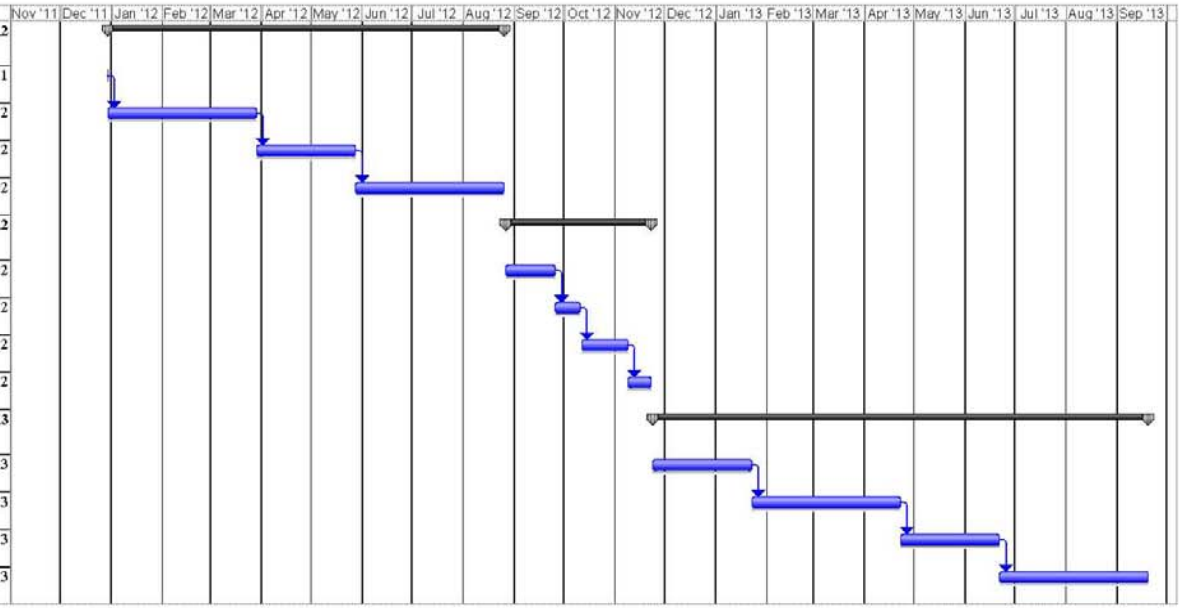
Analytical Group: Appendix IX VOCs, LLPAHs, Pesticides, and Metals; TPH GRO/DRO; RCRA Hazardous Waste Characteristics

Analyte	CAS No.	PAL (µg/L)	PAL Reference	Quantitation Goal (µg/L)	Laboratory-Specific Limits (µg/L)		
					LOQs	LODs	DLs
Cadmium	7440-43-9	NA	NA	1.00	1.00	0.500	0.140
Chromium	7440-47-3	NA	NA	2.00	2.00	1.00	0.140
Cobalt	7440-48-4	NA	NA	1.00	1.00	0.500	0.0400
Copper	7440-50-8	NA	NA	2.00	2.00	1.00	0.0620
Lead	7439-92-1	NA	NA	1.00	1.00	0.500	0.0600
Mercury (7470A)	7439-97-6	NA	NA	0.200	0.200	0.150	0.0355
Nickel	7440-02-0	NA	NA	1.00	1.00	0.500	0.0980
Selenium	7782-49-2	NA	NA	5.00	5.00	2.50	0.380
Silver	7440-22-4	NA	NA	1.00	1.00	0.500	0.0400
Thallium	7440-28-0	NA	NA	1.00	1.00	0.500	0.0430
Tin (6010C)	7440-31-5	NA	NA	50.0	50.0	25.0	2.87
Vanadium	7440-62-2	NA	NA	5.00	5.00	2.50	0.160
Zinc	7440-66-6	NA	NA	2.00	2.00	1.00	2.00
Total Petroleum Hydrocarbons (8015C)							
TPH DRO	9999-99-5	NA	NA	500	500	150	91
TPH GRO	9999-99-7	NA	NA	500	500	420	170
RCRA Hazardous Waste Characteristics							
Flashpoint/Ignitability (^{o}F) (ASTM D93-80)	NA	NA	NA	NA	NA	NA	NA
Reactive Cyanide (mg/L) (9014)	NA	NA	NA	125	125	NA	1.00
Reactive Sulfide (mg/L) (9034)	NA	NA	NA	125	125	NA	1.00
pH (S.U.) (9040C)	NA	NA	NA	NA	NA	NA	NA

SAP Worksheet #16: Project Schedule/Timeline

The schedule presented below is dependent upon regulator review time. Many other factors can also extend the schedule such as re-sampling if further re-characterization is required, weather delays in the field, or consensus cannot be reached on how the regulators comments are to be incorporated.

Task Name	Duration	Start	Finish
Sampling and Analysis Plan	**173 days**	**Fri 12/30/11**	**Sun 8/26/12**
Draft Sampling and Analysis Plan	0 edays	Fri 12/30/11	Fri 12/30/11
Regulatory Review	90 edays	Fri 12/30/11	Thu 3/29/12
Final Sampling and Analysis Plan	60 edays	Thu 3/29/12	Mon 5/28/12
Regulator Review and Approval	90 edays	Mon 5/28/12	Sun 8/26/12
Field Program Implementation	**65 days**	**Mon 8/27/12**	**Fri 11/23/12**
Project Initiation / Subcontractor Procurement	30 edays	Mon 8/27/12	Wed 9/26/12
Sampling and Data Collection	15 edays	Wed 9/26/12	Thu 10/11/12
Laboratory Analysis	28 edays	Fri 10/12/12	Fri 11/9/12
Data Validation	14 edays	Fri 11/9/12	Fri 11/23/12
Report Development	**214 days**	**Sat 11/24/12**	**Fri 9/20/13**
Draft Full RFI Report	60 edays	Sat 11/24/12	Wed 1/23/13
Regulatory Review	90 edays	Wed 1/23/13	Tue 4/23/13
Final Full RFI Report	60 edays	Tue 4/23/13	Sat 6/22/13
Regulator Review and Approval	90 edays	Sat 6/22/13	Fri 9/20/13



Project: SWMU 60 Full RFI
Date: Tue 12/20/11

Task Summary

SAP Worksheet #17: Sampling Design and Rationale

In order to achieve the PQOs for this Full RFI and support the decision-making process indentified on Worksheet #11, sampling and data collection activities will include a geophysical survey, surface and subsurface soil sampling, monitoring well installation and development, monitoring well abandonment and replacement, groundwater sampling, hydraulic conductivity testing, fluid level measurements, and sediment sampling. For investigation purposes, the SWMU has been divided into the following four areas based on history and site layout, potential source areas, release mechanisms, and contaminant distribution: (1) marina AST area, (2) suspected debris disposal areas, (3) SWMU-wide groundwater, and (4) Ensenada Honda. Details regarding the sampling design and rationale for each area are presented below. A summary of previous investigations and contaminant distribution was previously presented in the CSM (see Worksheet #10) and will not be reiterated herein.

17.1 Marina AST Area

Sampling and data collection activities proposed to be conducted at the marina AST area include surface/subsurface soil sampling and monitoring well abandonment/replacement as described below. Groundwater impacts will be addressed as part of the SWMU-wide groundwater program described in Section 17.3.

17.1.1 Surface and Subsurface Soil Sampling

Based on results from previous investigations, elevated VOCs, PAHs, arsenic, and TPH GRO and DRO were identified in surface and/or subsurface soil near the former underground piping system. Although the petroleum-impacted soil appears to be limited to the area between the ASTs and Ensenada Honda, the nature and extent of contamination has not been adequately delineated. As a result, six borings (60SB06 through 60SB11 as shown on Figure 10) will be advanced at this area. Borings 60SB06 through 60SB10 will be advanced at locations approximately 30 feet beyond wells 60SB04 and 60SB05. Due to the presence of the gasoline/diesel fuel ASTs, boring 60SB11 will be advanced at a location approximately 50 feet beyond well 60SB04. If petroleum-impacted soil is identified at an initial location based on PID measurements and/or visual/olfactory observations, an additional boring(s) will be advanced approximately 20 feet progressively farther outward from the impacted boring(s) until the extent of petroleum-impacted soil has been delineated. Conversely, if petroleum-impacted soil is not identified at an initial location based on field observations, an additional boring may be advanced approximately 15 feet farther inward from the initial boring to further refine the extent of impacted soil. The specific distance of the additional radially-spaced borings will be based on PID measurements, visual/olfactory observations, and/or professional judgment. For planning purposes, it is anticipated that four additional borings (60SB12 through 60SB15 as shown on Figure 10) may be required to complete the delineation. However, only those borings necessary to complete the delineation will be advanced (i.e., a boring may not be advanced at every location shown on Figure 10).

The borings will be advanced using a Geoprobe® rig (or equivalent) and direct push methods. Soil cores will be collected from each boring in 4-foot increments from the ground surface to the water table using disposable acetate liners and Macro-Core® samplers (or equivalent). Specific sample collection and field screening procedures are detailed on Worksheet #14 (Section 14.2.3).

One surface soil sample (0 to 1 foot bgs) and one shallow subsurface soil sample (1 to 3 feet bgs) will be collected from each boring for laboratory analysis. No deeper, subsurface soil samples are proposed due to the occurrence of shallow groundwater. However, if groundwater is encountered at depths greater than 3 feet bgs, one deeper soil sample will be collected from each boring at a depth shallower than observed groundwater. Specifically, these deeper soil samples will be collected from the depth interval exhibiting the highest PID measurement and/or visual/olfactory evidence of contamination. In lieu of any detected

PID measurements or visual/olfactory evidence of contamination, the deeper soil samples will be collected based on lithologic location (i.e., from the soil/groundwater interface) and/or professional judgment. Consideration will be given to these deeper sample intervals to ensure that representative data is collected for use in the HHRA that may be conducted as part of the CMS if warranted.
The samples will be analyzed for Appendix IX VOCs, LLPAHs, metals, and TPH GRO and DRO. Other organic constituents (e.g., SVOCs, pesticides, PCBs, herbicides) are not included in the list of analytes because they were not identified as COPCs based on review of historical data. Approximately 10% of the samples will also be analyzed for pH. The pH data will be used to calculate site-specific soil-to-groundwater screening values. The location-specific sampling method requirements are provided on Worksheet #18.1.

17.1.2 Monitoring Well Abandonment and Replacement

Two of the existing monitoring wells (60SB04 and 60SB05 as shown on Figure 10) installed during the Phase I RFI will be abandoned since the screened intervals are below the water table and the wells are not suitable to assess the presence/absence of LNAPL. Upon completion of the well abandonment, replacement wells will be installed adjacent to the initial wells. The replacement wells will be given an "R" designation (e.g., 60SB04R). Specific well abandonment and replacement procedures are detailed on Worksheet #14.

17.2 Suspected Debris Disposal Areas

Sampling and data collection activities proposed to be conducted at the suspected debris disposal areas include a geophysical survey and surface and subsurface soil sampling as described below. Groundwater impacts will be addressed as part of the SWMU-wide groundwater program described in Section 17.3.

17.2.1 Geophysical Survey

Based on results from previous investigations, the boundary limits of the three suspected debris disposal areas (1958 polygon features) shown on Figure 11 have not been adequately investigated. In addition, it is unknown whether buried debris exists at the SWMU. As a result, EM terrain conductivity and magnetic surveys will be conducted at each suspected disposal area by a qualified subcontractor. Data from this cursory investigation will be used in an attempt to: (1) define the boundary limits of the suspected disposal areas, (2) identify potential areas of buried debris if present, and (3) focus subsequent sampling and monitoring well installation activities.

EM instruments measure the electrical conductivity of subsurface materials by using a transmitter coil to generate an electromagnetic field that drives an electrical current into the ground. A receiver coil at a fixed separation from the transmitter is tuned and oriented to be insensitive to the transmitted field, and therefore measures only a secondary electromagnetic field generated by the subsurface electrical current. The strength and orientation of this field can be used to accurately estimate the bulk electrical conductivity (terrain conductivity) and relative ferrous and non-ferrous metallic content (in-phase response) of the surface/subsurface materials. Terrain conductivities are typically elevated in debris disposal areas due to the presence of exotic materials.

Magnetic surveys are sensitive to ferrous materials (e.g., iron or steel in tanks, drums, reinforcing, or debris) as well as materials that have been heated or burned at temperatures above their Curie point. Magnetometers measure the intensity of the earth's magnetic field along profiles or on a grid. In the absence of buried magnetic or magnetically susceptible objects (e.g., iron or steel containers, debris, burn residue, etc.), the intensity of the earth's magnetic field is generally constant or varies smoothly due to

variations in lithologic composition at depth. However, magnetic objects may create localized/abrupt variations or anomalies in the magnetic field intensity.

Each suspected debris disposal area will be thoroughly scanned along a series of transects using the EM terrain conductivity system followed by the magnetic system. The transects will be oriented in the east-west direction and will be spaced at approximate 50-foot intervals (see Figure 11). In addition, a transect oriented in the north-south direction will be scanned along the approximate centerline of each suspected disposal area. Suspected anomalous targets (i.e., surficial and/or buried debris) will be identified based on areas of higher-than-background values. Where suspected anomalous targets are detected, numerous more closely spaced and variously oriented transects will be scanned to more accurately determine the trace or footprint outline (and approximate depth where possible) of each target. The number, spacing, and orientation of additional transects will be determined in the field based on results from the initial/adjacent transects and professional judgment. The locations and depths (where possible) of any identified anomalies will be marked using wooden stakes and/or flagging and subsequently surveyed for inclusion on site mapping using conventional survey methods. In addition, any surficial anomalies will be visibly inspected to identify the nature of the anomalies (e.g., type of anomaly/debris; condition; presence of liquids, sludges, solids in containers). Specific geophysical survey procedures are detailed on Worksheet #14.

The geophysical surveying subcontractor will provide a report that documents the survey activities and results. The report will describe the geophysical procedures used and present figures depicting the locations of any identified anomalous targets. Back up information and associated interpretations of the data will also be provided as an appendix to the report.

Upon completion of the geophysical survey, the specific number and locations of proposed borings/monitoring wells within and near the suspected debris disposal areas will be discussed and agreed upon with the Navy, USEPA, and PREQB. It is anticipated that this will be accomplished via an on-site meeting and/or teleconference during the field program.

17.2.2 Surface and Subsurface Soil Sampling

Based on results from previous investigations, elevated PAHs, 4,4'-DDE, and metals were identified in surface and/or subsurface soil within the suspected debris disposal areas. However, the nature and extent of contamination has not been delineated. As a result, it is anticipated that approximately 17 borings (60SB16 through 60SB32) will be advanced within/near the three suspected disposal areas (see Figure 10). The anticipated number of borings is for planning purposes only. The specific number and locations of borings will be based on results of the geophysical survey described in Section 17.2.1 and contingent upon approval from the Navy, USEPA, and PREQB as described above. However, a sufficient number of borings will be advanced at locations adjacent to identified debris within the disposal areas to ensure that worst-case site conditions (i.e., areas posing the greatest potential risk to human health and the environment) will be captured/characterized. In addition, a sufficient number of borings will be advanced at locations outside (i.e., downgradient/sidegradient/upgradient) of the identified disposal areas to assist in characterization/delineation of potential contamination.

The borings will be advanced using a Geoprobe® rig (or equivalent) and direct push methods. Soil cores will be collected from each boring in 4-foot increments from the ground surface to the water table using disposable acetate liners and Macro-Core® samplers (or equivalent). Specific sample collection and field screening procedures are detailed on Worksheet #14.

Up to three soil samples will be collected from each boring as previously described in Section 17.1.1. The samples will be analyzed for Appendix IX VOCs, LLPAHs, pesticides, and metals. Although VOCs were not identified as COPCs in soil from the suspected disposal areas, they were included in the list of analytes to ensure that sufficient data is collected for use in the ERA and HHRA that may be conducted as part of the CMS if warranted. Other organic constituents (e.g., SVOCs, PCBs, herbicides) are not included in the list of analytes because they were not identified as COPCs based on review of historical data. Approximately 10% of the samples will also be analyzed for pH. The pH data will be used to calculate site-specific soil-to-groundwater screening values. The location-specific sampling method requirements are provided on Worksheet #18.2.

For boring locations in the wetland area, a determination will be made as to whether the sample is soil or sediment as previously described in Section 14.2.1. If significant sediments are present at a boring location, it will be noted in the field logbook(s), and the uppermost sample will be collected from 0 to 0.5 feet bgs (sediment depth interval) in lieu of 0 to 1 foot bgs (surface soil depth interval). In addition, TOC analysis will be added to the list of analytes for that sample.

17.3 SWMU-Wide Groundwater

Proposed sampling and data collection activities related to SWMU-wide groundwater include monitoring well installation and development, groundwater sampling, and hydraulic conductivity testing as described below. In addition, the interaction between shallow groundwater and Ensenada Honda will be evaluated.

17.3.1 Monitoring Well Installation and Development

Based on results of previous samples collected from existing monitoring wells at the SWMU, elevated VOCs, PAHs, and metals were identified in groundwater, and the nature and extent of contamination has not been delineated. As a result, it is anticipated that new monitoring wells will be installed at approximately five of the 27 boring locations used for soil sampling. The new wells will supplement the existing well network and facilitate collection of groundwater samples and determination of hydrogeologic conditions at the SWMU. One of the wells (60SB13 as shown on Figure 10) will be located in the presumed downgradient direction of the former underground piping system associated with the Marina's ASTs. It is anticipated that the remaining wells will be located within and downgradient of the suspected debris disposal areas to characterize worst-case groundwater conditions and delineate the extent of contamination. The specific number and locations of these wells will be based on results of the geophysical survey described in Section 17.2.1 and contingent upon approval from the Navy, USEPA, and PREQB.

Due to the occurrence of shallow groundwater, it is anticipated that the wells will be approximately 12 feet deep and installed such that the screens intersect the water table. Once a minimum of 24 hours has elapsed since installation, each new well will be developed using surging and bailing methods (or other suitable method) to remove fine-grained material and improve the hydraulic efficiency of the well. Specific well installation and development procedures are detailed on Worksheet #14.

17.3.2 Groundwater Sampling

Prior to groundwater sampling, a synoptic set of fluid level measurements will be collected from the new and existing monitoring wells at the SWMU and a number of existing wells at adjacent SWMUs/AOCs (e.g., JP-5 Hill and Diesel Fuel Marine Area of SWMU 74, AOC F 1995) to more accurately interpret the groundwater flow regime and any LNAPL thickness (if present).

Groundwater samples will be collected from the five new wells, two replacement wells (60SB04R and 60SB05R), and five existing wells (MW-1, MW-4, 6E-01, 6E-02, and 60SB02 as shown on Figure 10) after completion of well development and the water level has returned to equilibrium. A reasonable effort will be made to conduct the sampling after a minimum of 14 days has elapsed since completion of well development. However, this will be dependent on whether subsequent field activities are being conducted by Baker at other SWMUs/AOCs. In any case, the sampling will be planned accordingly such that the maximum time period between completion of well development and sampling is allowed. Groundwater data from the wells will be used to further characterize and determine the nature and extent of contamination.

If an existing well(s) cannot be located or has been damaged (i.e., well casing broken off), a decision will be made as to whether the well(s) needs replaced or can be repaired. The decision to replace a well will be based on the well's function in the monitoring network and whether sufficient data needs can be obtained from other wells in the network.

The groundwater samples will be collected using stainless steel bladder pumps and low flow purging/sampling methods to minimize sampling-induced turbidity problems and provide samples representative of ambient groundwater quality. Specific sampling procedures are detailed on Worksheet #14 and included in Appendix C.

The samples will be analyzed for Appendix IX VOCs, LLPAHs, pesticides, metals (total and dissolved fractions), and TPH GRO and DRO. Samples for dissolved metals will be filtered in the field prior to preservation using 0.45-micron disposable filters to eliminate turbidity and minimize suspended metals due to mobile colloidal particles. Although pesticides and TPH GRO and DRO were not identified as COPCs in groundwater, they are included in the list of analytes due to their presence in soil. Other organic constituents (e.g., SVOCs, PCBs, herbicides) are not included in the list of analytes because they were not identified as COPCs based on review of historical data. Samples from one of the existing wells (60SB04) and three of the new wells (60SB13 and two others TBD) will also be analyzed for TDS and TSS to provide an indication as to whether groundwater is suitable for use as potable water. The location-specific sampling method requirements are provided on Worksheet #18.3.

17.3.3 Hydraulic Conductivity Testing

Rising and/or falling head slug tests will be conducted at each new monitoring well to estimate the hydraulic conductivity of the saturated zone proximate to the wells. In addition, slug tests will be conducted at two of the existing wells (MW-1 and MW-4). Slug tests will not be conducted at the remaining three existing wells (6E-01, 6E-02, and 60SB02) since they were constructed as "temporary wells". Geologic/hydrogeologic information collected during well installation will be reviewed to ensure that the wells are representative of the various lithologies encountered at the SWMU. When possible, multiple tests will be conducted at a well to provide comparative data. Specific hydraulic conductivity testing and data analysis procedures are detailed on Worksheet #14.

17.3.4 Shallow Groundwater/Ensenada Honda Interaction

The interaction (i.e., tidal influence) between shallow groundwater and Ensenada Honda will be evaluated. As part of this evaluation, readily available information regarding construction of the retaining pier/boat docks (e.g., construction material, depth of penetration bgs) will be obtained through review of as-built drawings (if available) and interviews of NAPR personnel. In addition, salinity measurements will be collected from the new and existing monitoring wells at the SWMU during groundwater sampling.

Concurrent water levels will also be collected from Ensenada Honda, monitoring wells MW-4 and 60SB13, one of the new wells (TBD) located near the southwestern portion of the SWMU, and one of the new wells (TBD) located near the central or eastern portion of the SWMU. The water levels will be collected over a 72-hour period using computerized data loggers/pressure transducers. Specific data collection procedures are detailed on Worksheet #14.

Upon completion of the test, the data will be downloaded and processed using Microsoft Excel. The water level data, combined with information regarding construction of the retaining pier/boat docks and salinity measurements, will be evaluated to determine any interaction between shallow groundwater and Ensenada Honda and provide a more comprehensive understanding of the hydrogeologic conditions at the SWMU.

17.4 Ensenada Honda

Results from previous investigations indicated that surface water from Ensenada Honda was not impacted. Therefore, no surface water samples are proposed to be collected from Ensenada Honda as part of this Full RFI. However, elevated PAHs, 4,4'-DDE, and metals were identified in sediment (6E-SD01, 60SD01, and 60-SD02). Although the occurrence of these constituents likely is attributed to anthropogenic sources and not a release from the SWMU (see Worksheet #10), six sediment samples (60SD04 through 60SD09 as shown on Figure 10) will be collected from open water areas in Ensenada Honda to further characterize and determine the nature and extent of contamination in the vicinity of these sample locations. Samples 60SD04 through 60SD06 will be located approximately 100 feet radially outward (west, south, and east) from existing sample 60SD01 (Phase I RFI). Samples 60SD07 through 60SD09 will be located approximately 100 feet radially outward (north, west, and south) from existing sample 60SD02 (Phase I RFI).

The samples will be collected from 0 to 0.5 feet bgs using a decontaminated ponar dredge or sediment corer and analyzed for Appendix IX LLPAHs, pesticides, metals, and TOC. Other organic constituents (e.g., VOCs, SVOCs, PCBs, herbicides) are not included in the list of analytes because they were not identified as COPCs based on review of historical data. Specific sampling procedures are detailed on Worksheet #14, and location-specific sampling method requirements are provided on Worksheet #18.4.

SAP Worksheet #18.1: Location-Specific Sampling Method Requirements Table
Soil –Marina AST Area

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SB06	60-SB06-00 60-SB06-00D 60-SB06-00MS/MSD 60-SB06-01 60-SB06-XX	Soil	0.0-1.0 0.0-1.0 0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental 1 Field Duplicate 1 MS/MSD	Presumed Sidegradient; Further Characterize/Determine Nature and Extent of Contamination	See Worksheet #14
60SB07	60-SB07-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO; pH	3 Environmental	Presumed Downgradient; Further Characterize/Determine Nature and Extent of Contamination	
	60-SB07-01 60-SB07-XX		1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO			
60SB08	60-SB08-00 60-SB08-01 60-SB08-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental	Presumed Upgradient; Further Characterize/Determine Nature and Extent of Contamination	
60SB09	60-SB09-00 60-SB09-01 60-SB09-01D 60-SB09-01MS/MSD 60-SB09-XX	Soil	0.0-1.0 1.0-3.0 1.0-3.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental 1 Field Duplicate 1 MS/MSD	Presumed Downgradient; Further Characterize/Determine Nature and Extent of Contamination	
60SB10	60-SB10-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental	Presumed Upgradient; Further Characterize/Determine Nature and Extent of Contamination	
	60-SB10-01		1.0-3.0	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO; pH			
	60-SB10-XX		TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO			
60SB11	60-SB11-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO; pH	3 Environmental	Presumed Sidegradient; Further Characterize/Determine Nature and Extent of Contamination	
	60-SB11-01 60-SB11-XX		1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO			
60SB12	60-SB12-00 60-SB12-01 60-SB12-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental	Contingency Boring	
60SB13	60-SB13-00 60-SB13-01 60-SB13-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental	Contingency Boring; Probable Presumed Downgradient Well Location; Further Characterize/Determine Nature and Extent of Contamination	

SAP Worksheet #18.1: Location-Specific Sampling Method Requirements Table (*continued*)
Soil –Marina AST Area

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SB14	60-SB14-00 60-SB14-01 60-SB14-XX 60-SB14-XXD	Soil	0.0-1.0 1.0-3.0 TBD TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental 1 Field Duplicate	Contingency Boring	See Worksheet #14
60SB15	60-SB15-00 60-SB15-01 60-SB15-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, and Metals; TPH GRO/DRO	3 Environmental	Contingency Boring	

Notes:
XX – This indicates the designation for the depth interval from which the sample will be collected (i.e., 02 = 3.0-5.0 feet bgs, 03 = 5.0-7.0 feet bgs, etc.). The specific depth intervals will be established in the field based on PID measurements (highest measurement), visual/olfactory observations (staining/odors), lithologic location (soil/groundwater interface), and/or professional judgment.

Borings 60SB12 through 60SB15 have been included for contingency purposes and will only be advanced, if warranted, to delineate the extent of petroleum-impacted soil based on field observations.

SAP Worksheet #18.2: Location-Specific Sampling Method Requirements Table
Soil – Suspected Debris Disposal Areas

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SB16	60-SB16-00 60-SB16-00D 60-SB16-00MS/MSD 60-SB16-01 60-SB16-XX	Soil	0.0-1.0 0.0-1.0 0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental 1 Field Duplicate 1 MS/MSD	Within or Adjacent to (Radially Outward from) Suspected/Identified Debris Disposal Areas (see Notes); Further Characterize/Determine Nature and Extent of Contamination	See Worksheet #14
60SB17	60-SB17-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		
	60-SB17-01		1.0-3.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals, pH			
	60-SB17-XX		TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals			
60SB18	60-SB18-00 60-SB18-01 60-SB18-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		
60SB19	60-SB19-00 60-SB19-01 60-SB19-01D 60-SB19-XX	Soil	0.0-1.0 1.0-3.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental 1 Field Duplicate		
60SB20	60-SB20-00 60-SB20-01 60-SB20-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		
60SB21	60-SB21-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals, pH	3 Environmental		
	60-SB21-01 60-SB21-XX		1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals			
60SB22	60-SB22-00 60-SB22-01 60-SB22-XX 60-SB22-XXD	Soil	0.0-1.0 1.0-3.0 TBD TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental 1 Field Duplicate		
60SB23	60-SB23-00 60-SB23-01 60-SB23-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		

SAP Worksheet #18.2: Location-Specific Sampling Method Requirements Table (*continued*)
Soil – Suspected Debris Disposal Areas

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SB24	60-SB24-00 60-SB24-01 60-SB24-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental	Within or Adjacent to (Radially Outward from) Suspected/Identified Debris Disposal Areas (see Notes); Further Characterize/Determine Nature and Extent of Contamination	See Worksheet #14
60SB25	60-SB25-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		
	60-SB25-01		1.0-3.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals, pH			
	60-SB25-XX		TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals			
60SB26	60-SB26-00 60-SB26-00D 60-SB26-00MS/MSD 60-SB26-01 60-SB26-XX	Soil	0.0-1.0 0.0-1.0 0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental 1 Field Duplicate 1 MS/MSD		
60SB27	60-SB27-00 60-SB27-01 60-SB27-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		
60SB28	60-SB28-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals, pH	3 Environmental		
	60-SB28-01 60-SB28-XX		1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals			
60SB29	60-SB29-00 60-SB29-01 60-SB29-01D 60-SB29-XX	Soil	0.0-1.0 1.0-3.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental 1 Field Duplicate		
60SB30	60-SB30-00 60-SB30-01 60-SB30-XX	Soil	0.0-1.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental		

SAP Worksheet #18.2: Location-Specific Sampling Method Requirements Table (*continued*)
Soil – Suspected Debris Disposal Areas

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SB31	60-SB31-00	Soil	0.0-1.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental	Within or Adjacent to (Radially Outward from) Suspected/Identified Debris Disposal Areas (see Notes); Further Characterize/Determine Nature and Extent of Contamination	See Worksheet #14
	60-SB31-01		1.0-3.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals, pH			
	60-SB31-XX		TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals			
60SB32	60-SB32-00 60-SB32-01 60-SB32-01D 60-SB32-01MS/MSD 60-SB32-XX	Soil	0.0-1.0 1.0-3.0 1.0-3.0 1.0-3.0 TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals	3 Environmental 1 Field Duplicate 1 MS/MSD		

Notes:
The number of borings shown is for planning purposes only and will be based on results of the geophysical survey. Upon completion of the geophysical survey and evaluation of the data, the specific number and locations of borings will be discussed and agreed upon with the Navy, USEPA, and PREQB.

XX – This indicates the designation for the depth interval from which the sample will be collected (i.e., 02 = 3.0-5.0 feet bgs, 03 = 5.0-7.0 feet bgs, etc.). The specific depth intervals will be established in the field based on PID measurements (highest measurement), visual/olfactory observations (staining/odors), lithologic location (soil/groundwater interface), and/or professional judgment.

SAP Worksheet #18.3: Location-Specific Sampling Method Requirements Table
SWMU-Wide Groundwater

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
MW-1	MW01	Groundwater	1.5-11.5	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental	Existing Well; Presumed Upgradient of Marina AST Area/Downgradient of Suspected Debris Disposal Area; Further Characterize/Determine Nature and Extent of Contamination	See Worksheet #14 and Appendix C
MW-4	MW04	Groundwater	1.5-11.5	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental	Existing Well; Presumed Upgradient of Marina AST Area/Downgradient of Suspected Debris Disposal Area; Further Characterize/Determine Nature and Extent of Contamination	
6E-01	6E01	Groundwater	3.0-13.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental	Existing Well; Within Suspected Debris Disposal Area; Characterize Worst-Case Groundwater Conditions	
6E-02	6E02	Groundwater	3.0-13.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental	Existing Well; Within Suspected Debris Disposal Area; Characterize Worst-Case Groundwater Conditions	
60SB02	60GW02	Groundwater	2.0-5.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental	Existing Temporary Well; Within Suspected Debris Disposal Area; Characterize Worst-Case Groundwater Conditions	
60SB04R	60GW04R	Groundwater	5.5-15.5	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO; TDS/TSS	1 Environmental	Replacement Well; Along Alignment of Former Underground Piping System Associated with Marina AST Area; Characterize Worst-Case Groundwater Conditions	
60SB05R	60GW05R 60GW05RD 60GW05RMS/MSD	Groundwater	5.0-15.0	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental 1 Field Duplicate 1 MS/MSD	Replacement Well; Along Alignment of Former Underground Piping System Associated with Marina AST Area; Characterize Worst-Case Groundwater Conditions	
60SB13	60GW13	Groundwater	TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO; TDS/TSS	1 Environmental	New Well; Presumed Downgradient of Marina AST Area/Suspected Debris Disposal Area; Further Characterize/Determine Nature and Extent of Contamination	

SAP Worksheet #18.3: Location-Specific Sampling Method Requirements Table (*continued*)
SWMU-Wide Groundwater

Sampling Location/Identification Number		Matrix	Depth (feet bgs)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SBXX	60GWXX	Groundwater	TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental	New Wells; Within or Presumed Downgradient of Suspected/Identified Debris Disposal Areas (see Notes); Characterize Worst-Case Groundwater Conditions and/or Determine Nature and Extent of Contamination	See Worksheet #14 and Appendix C
60SBXX	60GWXX	Groundwater	TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO; TDS/TSS	1 Environmental		
60SBXX	60GWXX 60GWXXD	Groundwater	TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO	1 Environmental 1 Field Duplicate		
60SBXX	60GWXX	Groundwater	TBD	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total and dissolved fractions); TPH GRO/ DRO; TDS/TSS	1 Environmental		

Notes:
Wells MW-01, MW-04, 6E-01, 6E-02, and 60SB02 are existing wells and will be sampled if still present and determined to be usable.

The number of new wells shown is for planning purposes only and will be based on results of the geophysical survey. Upon completion of the geophysical survey and evaluation of the data, the specific number and locations of new wells will be discussed and agreed upon with the Navy, USEPA, and PREQB.

XX designation indicates that the specific boring for well installation has not been determined. This will be determined based on results of the geophysical survey as previously noted.

SAP Worksheet #18.4: Location-Specific Sampling Method Requirements Table
Sediment – Ensenada Honda

Sampling Location/Identification Number		Matrix	Depth (feet)	Analytical Group	Number of Samples	Rationale for Sampling Location	Sampling Reference
60SD04	60SD04 60SD04D 60SD04MS/MSD	Sediment	0.0-0.5	Appendix IX LLPAHs, Pesticides, and Metals	1 Environmental 1 Field Duplicate 1 MS/MSD	Radially Outward from Sample 60SD01 (Phase I RFI); Further Characterize/ Determine Nature and Extent of Contamination	See Worksheet #14
				TOC	1 Environmental		
60SD05	60SD05	Sediment	0.0-0.5	Appendix IX LLPAHs, Pesticides, and Metals; TOC	1 Environmental		
60SD06	60SD06	Sediment	0.0-0.5	Appendix IX LLPAHs, Pesticides, and Metals; TOC	1 Environmental		
60SD07	60SD07	Sediment	0.0-0.5	Appendix IX LLPAHs, Pesticides, and Metals; TOC	1 Environmental	Radially Outward from Sample 60SD02 (Phase I RFI); Further Characterize/ Determine Nature and Extent of Contamination	
60SD08	60SD08	Sediment	0.0-0.5	Appendix IX LLPAHs, Pesticides, and Metals; TOC	1 Environmental		
60SD09	60SD09	Sediment	0.0-0.5	Appendix IX LLPAHs, Pesticides, and Metals; TOC	1 Environmental		

SAP Worksheet #18.5: Location-Specific Sampling Method Requirements Table
Solid and Liquid IDW

Sampling Location/Identification Number		Matrix	Depth (feet)	Analytical Group	Number of Samples	Sampling Reference
Solid IDW	60IDW03	Soil	NA	TCLP VOCs, Pesticides, and Metals; Appendix IX LLPAHs; TPH GRO/DRO; Ignitability, Reactivity, and Corrosivity (pH)	1 Environmental	See Worksheet #14
Liquid IDW	60IDW04	Aqueous	NA	Appendix IX VOCs, LLPAHs, Pesticides, and Metals (total fraction); TPH GRO/DRO; Ignitability, Reactivity, and Corrosivity (pH)	1 Environmental	

SAP Worksheet #19: Field Sampling Requirements Table

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation/ analysis)
Soil	Appendix IX VOCs	5035A/8260B 1.1.4.1/1.3.2.4	Terra Core® 3 – 40 milliliter glass VOA vials; 1 – 2 ounce plastic	50 grams	1 vial with methanol; 2 vials with DI; store cool at 4° C	14 days
	Appendix IX LLPAHs	3550B/8270D-Selected Ion Monitoring (SIM) 2.5.2.3/2.4.4.5	1 – 8 ounce glass	100 grams	Store cool at 4° C	14 days
	Appendix IX Pesticides	3550B/8081B 2.2.4.2/2.2.4.10	1 – 8 ounce glass	8 ounces	Store cool at 4° C	14 days
	Appendix IX Metals	3050B/6020A/6010C (tin)/ 7471B (mercury) 3.2.1.5/3.2.1.9/3.2.1.6/3.3.4	1 – 2 ounce glass	1 gram	Store cool at 4° C	180 days; 28 days for mercury
	TPH GRO	5035A/8015C 1.1.4.1/1.1.3.2	Terra Core® 3 – 40 milliliter glass VOA vials; 1 – 2 ounce plastic	25 grams	1 vial with methanol; 2 vials with DI; store cool at 4° C	14 days
	TPH DRO	3550B/8015C 2.2.3.8/2.2.3.7	1 – 8 ounce glass	8 ounces	Store cool at 4° C	14 days
	pH	9045D 3.5.14.3	1 – 8 ounce glass	8 ounces	Store cool at 4° C	As soon as possible
Groundwater	Appendix IX VOCs	8260B 1.3.2.2	3 – 40 milliliter glass VOA vials	120 milliliters	HCl to pH<2; store cool at 4° C	14 days
	Appendix IX LLPAHs	3510C/8270D-SIM 2.5.2.1/2.4.4.5	2 – 1 liter glass amber	2 liters	Store cool at 4° C	7 days
	Appendix IX Pesticides	3510C/8081B 2.2.4.1/2.2.4.10	2 – 1 liter glass amber	2 liters	Store cool at 4° C	7 days
	Appendix IX Metals (Total)	3010A/6020A/6010C (tin)/ 7470A (mercury) 3.2.1.4/3.2.1.9/3.2.1.6/3.3.4	1 – 500 milliliter plastic	500 milliliters	HNO_3 to pH<2; store cool at 4° C	180 days; 28 days for mercury
	Appendix IX Metals (Dissolved)	3010A/6020A/6010C (tin)/ 7470A (mercury) 3.2.1.4/3.2.1.9/3.2.1.6/3.3.4	1 – 500 milliliter plastic (field-filtered)	500 milliliters	HNO_3 to pH<2; store cool at 4° C	180 days; 28 days for mercury
	TPH GRO	5030B/8015C 1.1.3.1	2 – 40 milliliter glass VOA vials	80 milliliters	HCl to pH<2; store cool at 4° C	14 days
	TPH DRO	3510C/8015C 2.2.3.1/2.2.3.7	2 – 1 liter glass amber	2 liters	Store cool at 4° C	7 days
	TDS	2540C 3.5.19.2	1 – 500 milliliter plastic	500 milliliters	Store cool at 4° C	7 days
	TSS	2540D 3.5.19.1	1 – 500 milliliter plastic	500 milliliters	Store cool at 4° C	7 days

SAP Worksheet #19: Field Sampling Requirements Table (*continued*)

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation/ analysis)
Sediment	Appendix IX LLPAHs	3550B/8270D-SIM 2.5.2.3/2.4.4.5	1 – 8 ounce glass	100 grams	Store cool at 4° C	14 days
	Appendix IX Pesticides	3550B/8081B 2.2.4.2/2.2.4.10	1 – 8 ounce glass	8 ounces	Store cool at 4° C	14 days
	Appendix IX Metals	3050B/6020A/6010C (tin)/ 7471B (mercury) 3.2.1.5/3.2.1.9/3.2.1.6/3.3.4	1 – 2 ounce glass	1 gram	Store cool at 4° C	180 days; 28 days for mercury
	TOC	Lloyd Kahn 3.6.2.2	1 – 4 ounce glass	4 ounces	Store cool at 4° C	14 days
Solid IDW	TCLP VOCs	1311/8260B 2.7.2/1.3.2.2	1 – 4 ounce glass	4 ounces	Store cool at 4° C	14 days to extraction; 14 days to analysis
	TCLP Pesticides	1311/8081B 2.7.2/2.2.4.3/2.2.4.10	1 – 8 ounce glass	100 grams	Store cool at 4° C	14 days to extraction; 7 days to preparative extraction; 40 days to analysis
	TCLP Metals	1311/6010C/7470A (mercury) 2.7.2/3.2.1.6/3.3.4	1 – 8 ounce glass	100 grams	Store cool at 4° C	180 days to extraction; 180 days to analysis; mercury-28 days to extraction; 28 days to analysis
	Appendix IX LLPAHs	3550B/8270D-SIM 2.5.2.3/2.4.4.5	1 – 8 ounce glass	100 grams	Store cool at 4° C	14 days
	TPH GRO	5035A/8015C 1.1.4.1/1.1.3.2	Terra Core® 3 – 40 milliliter glass VOA vials; 1 – 2 ounce plastic	25 grams	1 vial with methanol; 2 vials with DI; store cool at 4° C	14 days
	TPH DRO	3550B/8015C 2.2.3.8/2.2.3.7	1 – 8 ounce glass	8 ounces	Store cool at 4° C	14 days
	Ignitability, Reactivity, Corrosivity (pH)	ASTM D93-80/9014/9034/9040C 3.5.9.1/3.4.4/3.5.18.1/3.5.14.2	1 – 8 ounce glass	8 ounces	Store cool at 4° C	As soon as possible

SAP Worksheet #19: Field Sampling Requirements Table (*continued*)

Note:

Matrix	Analytical Group	Preparation and Analytical Method/SOP Reference	Containers (number, size, and type)	Sample Volume	Preservation Requirements (chemical, temperature, light protected)	Maximum Holding Time[1] (preparation/ analysis)
Liquid IDW	Appendix IX VOCs	8260B 1.3.2.2	3 – 40 milliliter glass VOA vials	120 milliliters	HCl to pH<2; store cool at 4° C	14 days
	Appendix IX LLPAHs	3510C/8270D-SIM 2.5.2.1/2.4.4.5	2 – 1 liter glass amber	2 liters	Store cool at 4° C	7 days
	Appendix IX Pesticides	3510C/8081B 2.2.4.1/2.2.4.10	2 – 1 liter glass amber	2 liters	Store cool at 4° C	7 days
	Appendix IX Metals (Total)	3010A/6020A/6010C (tin)/ 7470A (mercury) 3.2.1.4/3.2.1.9/3.2.1.6/3.3.4	1 – 500 milliliter plastic	500 milliliters	HNO_3 to pH<2; store cool at 4° C	180 days; 28 days for mercury
	TPH GRO	5030B/8015C 1.1.3.1	2 – 40 milliliter glass VOA vials	80 milliliters	HCl to pH<2; store cool at 4° C	14 days
	TPH DRO	3510C/8015C 2.2.3.1/2.2.3.7	2 – 1 liter glass amber	2 liters	Store cool at 4° C	7 days
	Ignitability, Reactivity, Corrosivity (pH)	ASTM D93-80/9014/9034/9040C 3.5.9.1/3.4.4/3.5.18.1/3.5.14.2	2 – 1 liter glass ambers	2 liters	Store cool at 4° C	As soon as possible

1 - Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

SAP Worksheet #20: Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Rinsate Blanks	No. of Trip Blanks	No. of Proficiency Testing (PT) Samples	Total No. of Samples to Laboratory
Soil	Appendix IX VOCs	81	9	5	2	5	5	0	107
	Appendix IX LLPAHs	81	9	5	2	5	0	0	102
	Appendix IX Pesticides	51	6	3	2	5	0	0	67
	Appendix IX Metals	81	9	5	2	5	0	0	102
	TPH GRO	30	3	2	2	5	2	0	44
	TPH DRO	30	3	2	2	5	0	0	42
	pH	8	0	0	0	0	0	0	8
Groundwater	Appendix IX VOCs	12	2	1	0	3	3	0	21
	Appendix IX LLPAHs	12	2	1	0	3	0	0	18
	Appendix IX Pesticides	12	2	1	0	3	0	0	18
	Appendix IX Metals (Total)	12	2	1	0	3	0	0	18
	Appendix IX Metals (Dissolved)	12	2	1	0	3	0	0	18
	TPH GRO	12	2	1	0	3	3	0	21
	TPH DRO	12	2	1	0	3	0	0	18
	TDS	4	0	0	0	0	0	0	4
	TSS	4	0	0	0	0	0	0	4

SAP Worksheet #20: Field Quality Control Sample Summary Table (*continued*)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Rinsate Blanks	No. of Trip Blanks	No. of PT Samples	Total No. of Samples to Laboratory
Sediment	Appendix IX LLPAHs	6	1	1	0	2	0	0	10
	Appendix IX Pesticides	6	1	1	0	2	0	0	10
	Appendix IX Metals	6	1	1	0	2	0	0	10
	TOC	6	0	0	0	0	0	0	6
Solid IDW	TCLP VOCs	1	0	0	0	0	0	0	1
	TCLP Pesticides	1	0	0	0	0	0	0	1
	TCLP Metals	1	0	0	0	0	0	0	1
	Appendix IX LLPAHs	1	0	0	0	0	0	0	1
	TPH GRO	1	0	0	0	0	0	0	1
	TPH DRO	1	0	0	0	0	0	0	1
	Ignitability	1	0	0	0	0	0	0	1
	Reactivity	1	0	0	0	0	0	0	1
	Corrosivity	1	0	0	0	0	0	0	1
Liquid IDW	Appendix IX VOCs	1	0	0	0	0	0	0	1
	Appendix IX LLPAHs	1	0	0	0	0	0	0	1
	Appendix IX Pesticides	1	0	0	0	0	0	0	1

SAP Worksheet #20: Field Quality Control Sample Summary Table (*continued*)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates	No. of MS/MSDs	No. of Field Blanks	No. of Equipment Rinsate Blanks	No. of Trip Blanks	No. of PT Samples	Total No. of Samples to Laboratory
Liquid IDW (*continued*)	Appendix IX Metals (Total)	1	0	0	0	0	0	0	1
	TPH GRO	1	0	0	0	0	0	0	1
	TPH DRO	1	0	0	0	0	0	0	1
	Ignitability	1	0	0	0	0	0	0	1
	Reactivity	1	0	0	0	0	0	0	1
	Corrosivity	1	0	0	0	0	0	0	1

SAP Worksheet #21: Project Sampling SOP References Table

Reference Number	Title, Revision Date and/or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
Groundwater Sampling SOP	Groundwater Sampling Procedure, Low Stress (Low Flow) Purging and Sampling, March 16, 1998	USEPA Region II	Groundwater Sampling Pumps and Tubing	N	See Appendix C for SOP

Note:
See Worksheet #14 for detailed sampling and data collection procedures associated with this SAP.

SAP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
GPS Unit	No calibration required	Recharge battery daily; clean meter housing with a damp, lint-free cloth	Visual inspection; check battery	Before use each day	Accuracy: sub-meter; horizontal dilution of precision (HDOP): <3; number of satellites: at least six; real time correction: on	Wait for better satellite constellation, replace unit, or choose alternate location technique	Jason Oliver/ FOL
PID Meter - MiniRae 2000	Calibrate using ambient air and isobutylene 100 parts per million (ppm) calibration gas	Recharge battery daily; clean meter housing with a damp, lint-free cloth; clean sensor module, lamp, and lamp housing as warranted	Visual inspection; check battery	Before use each day; when unstable or erroneous readings occur	Ambient air reads 0.0 ppm +/-3%; isobutylene gas reads 100 ppm +/-3%	Clean sensor module, lamp, and lamp housing and re-calibrate; replace if unable to calibrate properly	
Water Level Indicator	No calibration required	Clean meter housing, tape, and probe; replace battery as warranted	Visual inspection; check battery	Before use each day; between each well	Maintained in good working order per manufacturer's recommendations	Replace if not working properly	
Oil/Water Interface Probe	No calibration required	Clean meter housing, tape, and probe; replace battery as warranted	Visual inspection; check battery	Before use each day; between each well	Maintained in good working order per manufacturer's recommendations	Replace if not working properly	
Water Quality Meter - YSI 556 MPS	Calibrate probe using multiple calibration standard solutions provided	Verify system continuity; clean meter housing with a damp, lint-free cloth; clean probe with DI water; replace dissolved oxygen membrane and batteries as warranted	Visual inspection; check batteries	Before use each day; when unstable or erroneous readings occur	pH, conductivity, and ORP reads +/-3% of the calibration solution values; dissolved oxygen reading is consistent with the current atmospheric pressure and ambient temperature	Clean probe, replace dissolved oxygen membrane, and re-calibrate; replace if unable to calibrate properly	
Turbidity Meter	Calibrate using 1 and/ or 10 Nephelometric Turbidity Unit (NTU) standard solutions	Clean meter housing with a damp, lint-free cloth; clean meter chamber and lenses with a lint-free cloth slightly dampened with alcohol and replace battery as warranted	Visual inspection; check battery	Before use each day; when unstable or erroneous readings occur	Turbidity reads +/-3% of the calibration solution values	Clean meter chamber and lenses and re-calibrate; replace if unable to calibrate properly	
QED Sample Pro Bladder Pump, Controller, and Compressor	No calibration required	Disassemble pump and clean components; replace bladder; clean controller and compressor housing with a damp, lint-free cloth	Inspect components, tubing, and air/sample line quick-connects	Before use each day; between each well	Maintained in good working order per manufacturer's recommendations	Replace if not working properly	
Data Logger/ Pressure Transducer - AquaStar PT2X Smart Sensor	No calibration required	Clean probe and cable; replace batteries and desiccant tube as warranted	Inspect probe, cable, and end connections	Before use each day; between each well	Maintained in good working order per manufacturer's recommendations	Replace if not working properly	

SAP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table (*continued*)

Field Equipment	Calibration Activity	Maintenance Activity	Testing/Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
Geonics EM-31 MKII Electromagnetic Terrain Conductivity Meter	Cable shake test; direct current level zeroing; compensation and phasing set per manufactures instructions	Replace batteries when meter indicates 80% of full charge	Visual inspection; check batteries	Before use each day	Direct current level at zero; compensation at zero; phasing at zero	Recalibrate in electromagnetically clean location	Geophysics Project Manager
Juniper Systems Allegro Data Logger	No calibration required	Recharge battery daily; Recharge data logger daily	Visual inspection; check battery	Before use each day	Position dilution of precision (PDOP) mask: 8; signal to noise ratio (SNR) mask: 10; real time correction: on	Wait for better satellite constellation; switch to gridded survey	
Geometrics G-858g MagMapper magnetic gradiometer	Cable shake test; azimuth test over 2-inch hitch ball; six line test over 2-inch hitch ball; walk away noise test; operator cleanliness test	Recharge batteries daily	Visual inspection; check batteries	Before use each day	Noise level <2 nT; 2-inch hitch ball anomaly >20 nT at 18 inches	Change sensor	
Topcon GPS with integrated beacon and wide area augmentation system (WAAS) and handheld data logger	No calibration required	Recharge battery daily; Recharge data logger daily	Visual inspection; check battery	Before use each day	PDOP mask: 8; SNR mask: 10; real time correction: on	Wait for better satellite constellation; switch to gridded survey	

Note:
Specific instructions and user manuals will be provided with each piece of equipment.

SAP Worksheet #23: Analytical SOP References Table

Laboratory SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
1.1.4.1	Preparation of Soil/Sediment/Sludge/Non-Aqueous Waste Samples for the Analysis of VOCs by Closed-System Purge and Trap by SW-846 Methods 5035A and 3585 and EPA Contract Laboratory Program (CLP) Statement of Work SOM01.2	NA	Soil/VOCs	NA	CompuChem	N
1.3.2.2	Analysis of VOCs in Aqueous and Medium/High Concentration Soil Samples by SW-846; Revision 15; April 11, 2011	Definitive	Soil, Groundwater/VOCs	GC/MS	CompuChem	N
1.3.2.4	Gas Chromatograph/Mass Spectrometer (GC/MS) Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 Method 8260B; Revision 13; June 20, 2011	Definitive	Soil/VOCs	GC/MS	CompuChem	N
2.5.2.3	Preparation of Soil/Sediment/Sludge Samples by SW-846 Method 3550B and Method 3550C for the analysis of Low-Level Semivolatiles; Revision 16; April 17, 2012	Definitive	Soil, Sediment/LLPAHs	NA	CompuChem	N
2.5.2.1	Preparation of Water Samples for the Analysis of Semivolatiles by SW-846 Method 3510C; Revision 15; October 25, 2011	Definitive	Groundwater/LLPAHs	NA	CompuChem	N
2.4.4.5	GC/MS SIM Semivolatile Analyses of Aqueous and Soil Samples using SW-846; Revision 3; May 18, 2011	Definitive	Soil, Groundwater, Sediment/LLPAHs	GC/MS	CompuChem	N
2.2.4.2	Low Level Preparation for Analysis of Pesticides/PCBs in Soil/Sediment/Sludge by SW-846 Methods 3550B and 3550C; Revision 13; April 27, 2011	Definitive	Soil, Sediment/Pesticides	NA	CompuChem	N
2.2.4.1	Sample Preparation for Pesticides/PCBs in Water by SW-846; Revision 14; October 27, 2011	Definitive	Groundwater/Pesticides	NA	CompuChem	N
2.2.4.10	GC/Electron Capture Detection (ECD) Analysis of Organochlorine Pesticides in Water and Soil Extracts by SW-846 Method 8081B; Revision 9; April 11, 2011	Definitive	Soil, Groundwater, Sediment/Pesticides	GC/ECD	CompuChem	N
3.2.1.5	Digestion Block Preparation of Solid Samples for Inductively Coupled Plasma (ICP) Determination of Total Metals by SW-846 Method 3050B; Revision 8; January 12, 2011	Definitive	Soil, Sediment/Metals	NA	CompuChem	N
3.2.1.4	Digestion Block Preparation of Aqueous Samples for ICP Analysis of Total or Dissolved Metals by SW-846 and Standard Methods; Revision 5; January 10, 2011	Definitive	Groundwater/Metals	NA	CompuChem	N
3.2.1.6	Inductively Coupled Plasma Atomic Emission Spectroscopy (AES) by SW-846 Methods 6010B and 6010C; Revision 19; October 17, 2011	Definitive	Soil, Groundwater, Sediment/Metals	ICP-AES	CompuChem	N

SAP Worksheet #23: Analytical SOP References Table (*continued*)

Laboratory SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
3.2.1.9	Determination of Metals by ICP-MS by SW-846 Method 6020/6020A; Revision 6; October 18, 2011	Definitive	Soil, Groundwater, Sediment/Metals	ICP-MS	CompuChem	N
3.3.4	Automated Cold Vapor Determination for Mercury by CLP, SW-846, and Methods for Chemical Analysis of Water and Wastes (MCAWW); Revision 24; April 6, 2011	Definitive	Soil, Groundwater, Sediment/Metals	Cold Vapor Atomic Adsorption (CVAA)	CompuChem	N
1.1.3.1	Preparation and GC Analysis of GRO in Aqueous Samples by Purge and Trap Following SW-846, California Leaking Underground Fuel Tanks (CA LUFT), and Tennessee GRO; Revision 13; February 16, 2011	Definitive	Groundwater/TPH GRO	GC/Flame Ionization Detector (FID)	CompuChem	N
1.1.3.2	Preparation and Analysis of GRO in Soil/ Sediment Samples by Purge and Trap Following SW-846, CA LUFT, and Tennessee GRO; Revision 11; February 1, 2011	Definitive	Soil/TPH GRO	GC/FID	CompuChem	N
2.2.3.8	Preparation of Soil/Sediment/Sludge Samples for DRO and Oil Range Organics by SW-846 Method 3550B, Method 3550C, and DRO by Tennessee Department of Environment and Conservation; Revision 4; October 21, 2011	Definitive	Soil/TPH DRO	NA	CompuChem	N
2.2.3.1	Preparation of Water Samples for DRO and Oil Range Organics by SW-846 Method 3510C and Tennessee Department of Environment and Conservation; Revision 12; April 27, 2011	Definitive	Groundwater/TPH DRO	NA	CompuChem	N
2.2.3.7	Analysis of DRO, Oil Range Organics, JP4, JP8, and Total Jet Fuel in Aqueous and Solid Samples by SW-846 and Extractable Petroleum Hydrocarbons by Tennessee Department of Environment and Conservation; Revision 12; June 3, 2010	Definitive	Soil, Groundwater/TPH DRO	GC/FID	CompuChem	N
3.5.19.2	Filterable Residue, TDS, by Standard Methods for the Examination of Water and Wastewater, Revision 8, July 15, 2010	Definitive	Groundwater/TDS	Gravimetric	CompuChem	N
3.5.19.1	Non-filterable Residue, TSS, by Standard Methods for the Examination of Water and Wastewater, Revision 11, September 1, 2010	Definitive	Groundwater/TSS	Gravimetric	CompuChem	N
3.6.2.2	Analysis of Soil Samples for TOC by SW-846 Method 9060A, modified for Soils, and Lloyd Kahn; Revision 16 ; Feb 1, 2011	Definitive	Sediment/TOC	Non-Dispersive Infrared Detector (NDIRD)	CompuChem	N
2.7.2	TCLP by SW-846 Method 1311	NA	Solid IDW/TCLP VOCs, Pesticides, Metals	NA	CompuChem	N

SAP Worksheet #23: Analytical SOP References Table (*continued*)

Laboratory SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
2.2.4.3	Extraction of TCLP Leachates for the Determination of Pesticides/PCBs by SW-846	NA	Solid IDW/TCLP Pesticides	NA	CompuChem	N
3.2.16	ICP-AES by SW-846 Methods 6010B and 6010C; Revision 19; October 17, 2011	Definitive	Solid IDW/TCLP Metals	ICP-AES	CompuChem	N
3.3.4	Automated Cold Vapor Determination for Mercury by CLP, SW-846, and MCAWW; Revision 24; April 6, 2011	Definitive	Solid IDW/TCLP Metals	CVAA	CompuChem	N
3.5.9.1	Ignitability by SW-846 Method 1010A and American Society for Testing and Materials (ASTM) D93-80 (Pensky-Martens); Revision 6; June 14, 2011	Definitive	Solid IDW, Liquid IDW/Ignitability	Pensky-Martens Closed Cup Tester	CompuChem	N
3.4.4	Reactive (Total Releasable) Cyanide by SW-846; Revision 8; November 24, 2010	Definitive	Solid IDW, Liquid IDW/Reactivity	Titration	CompuChem	N
3.5.18.1	Reactive (Total Releasable) Sulfide by SW-846 Chapter 7, Section 7.3.4 and Method 9034; Revision 8; June 3, 2011	Definitive	Solid IDW, Liquid IDW/Reactivity	Titration	CompuChem	N
3.5.14.2	Corrosivity Characterization by pH Determination in SW-846; Revision 6; June 28, 2011	Definitive	Solid IDW, Liquid IDW/Corrosivity	Electrode	CompuChem	N
3.5.14.3	Soil and Waste pH by SW-846 Methods 9045C and 9045D; Revision 5; January 14, 2011	Definitive	Soil/pH	Electrode	CompuChem	N
4.1	Receiving Samples; Revision 36; April 04, 2011	NA	Soil, Groundwater, Sediment, Solid IDW, Liquid IDW	NA	CompuChem	N
12.1	Hazardous Waste Disposal; Revision 8; August 18, 2010	NA	Soil, Groundwater, Sediment, Solid IDW, Liquid IDW	NA	CompuChem	N

SAP Worksheet #24: Analytical Instrument Calibration Table

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/MS (VOA)	Tune Verification	The tune is verified at the beginning of each 12-hour analytical sequence	The tune verification must meet the ion abundance criteria required by the method	Manually tune the instrument and/or replace the ion source or filament	Analyst	1.3.2.2 1.3.2.4
	Initial Calibration (ICAL)	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification	The average Response Factor (RF) for VOC System Performance Check Compounds (SPCCs) must be ≥0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane; the Relative Standard Deviation (RSD) for Calibration Check Compounds (CCCs) for VOCs must be ≤ 30%; RSD for each analyte must be ≤ 15% or the linear least squares regression (r) must be ≥ 0.995	Recalibrate		1.3.2.2 1.3.2.4
	ICAL Verification	Perform after each initial calibration	The percent recovery of all analytes must be 80-120%	Recalibrate		1.3.2.2 1.3.2.4
	Continuing Calibration Verification (CCV)	Perform one per 12-hour analysis period	The RF for VOC SPCCs must be ≥0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane; the percent difference/drift must be ≤ 20%	Recalibrate		1.3.2.2 1.3.2.4
GC/MS Semivolatile Organic Analytes (SVOA)	Tune Verification	The tune is verified at the beginning of each 12-hour analytical sequence	Tailing factor for benzidine and pentachlorophenol ≤ 2.0; DDT breakdown ≤ 20%	Manually tune the instrument and/or replace the ion source or filament	Analyst	2.4.4.5
	ICAL	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification	The average RF must be ≥ 0.010; RSD for each analyte must be ≤ 15% or the linear least squares regression (r) must be ≥ 0.995	Recalibrate		2.4.4.5
	ICAL Verification	Perform after each initial calibration	The percent recovery of all analytes must be 80-120%	Recalibrate		2.4.4.5
	CCV	Perform one per 12-hour analysis period	The RF must be ≥ 0.010; the percent difference/drift must be ≤ 20%	Recalibrate		2.4.4.5
GC/ECD	Breakdown Check (Endrin/DDT)	Perform one per 12-hour analysis period, prior to analysis of samples	Degradation ≤ 15% for both DDT and Endrin	Injector port maintenance and/or column maintenance or replacement	Analyst	2.2.4.10
	ICAL	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification	RSD for each analyte must be ≤ 20% or the linear least squares regression (r) must be ≥ 0.995	Recalibrate		2.2.4.10

SAP Worksheet #24: Analytical Instrument Calibration Table (*continued*)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
GC/ECD	ICAL Verification	Perform after each initial calibration	The percent recovery of all analytes must be 80-120%	Recalibrate	Analyst	2.2.4.10
	CCV	Perform prior to sample analysis, after each 10 samples, and at the end of the analysis sequence	The percent difference/drift must be ≤ 20%	Recalibrate		2.2.4.10
ICP-AES	ICAL	Perform each analytical sequence	There are no criteria; only one standard is analyzed	Recalibrate	Analyst	3.2.1.6
	ICAL Verification	Perform each analytical sequence	The percent recovery of all analytes must be 90-110%	Recalibrate		3.2.1.6
	ICAL Blank (ICB)	Perform each analytical sequence	The concentration of the target analytes must be ≤ the LOD	Recalibrate		3.2.1.6
	CCV	Perform after each 10 samples and at the end of the analysis sequence	The percent recovery of all analytes must be 90-110%	Recalibrate		3.2.1.6
	Continuing Calibration Blank (CCB)	Perform after each 10 samples and at the end of the analysis sequence	The concentration of the target analytes must be ≤ the LOD	Recalibrate		3.2.1.6
	Interference Check Solution (ICS)-A and ICS-AB)	Perform at the beginning of an analytical run and every 12 hours	ICS-A: absolute value of the concentration for all non-spiked analytes < LOD; ICS-AB: within ± 20% of true value	Recalibrate		3.2.1.6
ICP-MS	Tune Verification	Perform each analytical sequence	Resolution within 0.1 Atomic Mass Units (AMU) of target mass at 10% peak height and %RSD of five replicates are less than 5	Perform Tune	Analyst	3.2.1.9
	ICAL	Perform each analytical sequence	Correlation coefficient of curve must be ≥ 0.998	Recalibrate		3.2.1.9
	ICAL Verification	Perform each analytical sequence	The percent recovery of all analytes must be 90-110%	Recalibrate		3.2.1.9
	ICB	Perform each analytical sequence	The concentration of the target analytes must be ≤ the LOD	Recalibrate		3.2.1.9
	CCV	Perform after each 10 samples and at the end of the analysis sequence	The percent recovery of all analytes must be 90-110%	Recalibrate		3.2.1.9
	CCB	Perform after each 10 samples and at the end of the analysis sequence	The concentration of the target analytes must be ≤ the LOD	Recalibrate		3.2.1.9
	ICS-A and ICS-AB	Perform at the beginning of an analytical run and every 12 hours	ICS-A: absolute value of the concentration for all non-spiked analytes < LOD; ICS-AB: within ± 20% of true value	Recalibrate		3.2.1.9

SAP Worksheet #24: Analytical Instrument Calibration Table (*continued*)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
CVAA	ICAL	Perform each analytical sequence	Correlation coefficient of curve must be ≥ 0.995	Recalibrate	Analyst	3.3.4
	ICAL Verification	Perform each analytical sequence	The percent recovery must be 90-110%	Recalibrate		3.3.4
	ICB	Perform each analytical sequence	The concentration of the target analytes must be ≤ the LOD	Recalibrate		3.3.4
	CCV	Perform after each 10 samples and at the end of the analysis sequence	The percent recovery must be 80-120%	Recalibrate		3.3.4
	CCB	Perform after each 10 samples and at the end of the analysis sequence	The concentration of the target analytes must be ≤ the LOD	Recalibrate		3.3.4
GC/FID (GRO)	ICAL	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification	RSD for each analyte must be ≤ 20% or the linear least squares regression (r) must be ≥ 0.995	Recalibrate	Analyst	1.1.3.1 1.1.3.2
	ICAL Verification	Perform after each initial calibration	The percent recovery must be 80-120%	Recalibrate		1.1.3.1 1.1.3.2
	CCV	Perform prior to sample analysis, after each 10 samples and at the end of the analysis sequence	The percent difference/drift must be ≤ 15%	Recalibrate		1.1.3.1 1.1.3.2
GC/FID (DRO)	ICAL	Perform after major instrument maintenance and upon failure of second consecutive continuing calibration verification	RSD for each analyte must be ≤ 20% or the linear least squares regression (r) must be ≥ 0.995	Recalibrate	Analyst	2.2.3.7
	ICAL Verification	Perform after each initial calibration	The percent recovery must be 80-120%	Recalibrate		2.2.3.7
	CCV	Perform prior to sample analysis, after each 10 samples and at the end of the analysis sequence	The percent difference/drift must be ≤ 20%	Recalibrate		2.2.3.7
NDIRD	ICAL	Perform after major instrument maintenance OR upon failure of second consecutive c initial calibration verification	Correlation coefficient of curve must be ≥ 0.995	Recalibrate	Analyst	3.6.2.2
	ICAL Verification	Perform each analytical sequence	The percent recovery must be 90-110%	Recalibrate		3.6.2.2

SAP Worksheet #24: Analytical Instrument Calibration Table (*continued*)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference
NDIRD	CCV	Perform after each 10 samples and at the end of the analysis sequence	The percent recovery must be 90-110%	Recalibrate	Analyst	3.6.2.2
Pensky-Martens Closed Cup Tester	ICAL Verification	Perform each analytical sequence	Acceptable criteria is within 2° F of known flashpoint value	Re-analyze	Analyst	3.5.9.1
Electrode	ICAL	Perform each analytical sequence	Efficiency between 90 and 105% and calculated values of pH solution within +/- 0.05 pH units	Recalibrate	Analyst	3.5.14.2
	ICAL Verification	Perform each analytical sequence	Acceptable criteria is determined by vendor and listed on the COA	Recalibrate		3.5.14.2
	CCV	Perform after each 10 samples and at the end of the analysis sequence	Acceptable criteria is within +/-0.10 pH unit of true value of buffer solution	Recalibrate		3.5.14.2
Balance	Verify weight calibration using National Bureau of Standards/National Institute of Standards and Technology Class "S" weights	Daily	Weight reading within tolerance range specified in SOP 13.17	Repeat activity or remove from service	Analyst	3.5.19.1 and 3.5.19.2

SAP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GC/MS	Replace or clean ion source; clean injector; replace injector liner; replace or clip capillary column; flush or replace tubing on purge and trap; replace trap	VOCs/SVOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service	Laboratory Area Supervisor/Analyst	1.3.2.2 1.3.2.4 2.4.4.5
GC/ECD	ECD maintenance; replace/clip capillary column	Pesticides	ECD, injector, injector liner, column, column flow	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service		2.2.4.10
ICP-AES	Clean plasma torch; clean filters; clean spray and nebulizer chambers; replace pump tubing	Metals	Torch, filters, nebulizer chamber, pump, pump tubing	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service		3.2.1.6
ICP-MS	Replace torch; clean filters; clean spray and nebulizer chambers; clean or replace cones; replace autolen; replace pump tubing	Metals	Torch, filters, nebulizer chamber, pump, pump tubing; cones	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service		3.2.1.9
CVAA	Clean or replace dehydrator tubing and sample mixing coil tubing; replace sample probe; replace pump tubing; clean optical cell	Mercury	Tubing, sample probe, optical cell	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service		3.3.4
GC/FID	FID maintenance; replace/clip capillary column	DRO/GRO	FID, injector, injector liner, column, column flow	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service		1.1.3.1 1.1.3.2 2.2.3.7
NDIRD	Replace sample tubing; clean sample boat; replace syringe	TOC	Tubing, sample boat, syringe	Perform as needed	Initial and/or continuing calibration criteria must be met	Repeat maintenance activity or remove from service		3.6.2.2

SAP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table (*continued*)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
Pensky-Martens Closed Cup Tester	Lid tightness, shutter action; flame position; propane level	Ignitability	Lid, shutter, flame	Perform as needed	Initial calibration criteria must be met	Repeat maintenance activity or remove from service	Laboratory Area Supervisor/Analyst	3.5.9.1
Electrode	Clean probe	pH	Probe	Perform as needed	The value for each of the certified buffer solutions must be within ± 0.05 pH units of the expected value	Repeat maintenance activity or remove from service		3.5.14.2
Balance	Clear of debris and soil sample spillage	TDS/TSS	Verify that all calibration weights are in good condition and have been calibrated within the last 12 months; verify that balance calibration has been performed within the last 6 months and is certified with a sticker	Perform as needed	Weight reading within tolerance range specified in SOP 13.17	Repeat maintenance activity or remove from service		3.5.19.2 3.5.19.1 13.17
Oven	Adjust oven set point	TDS/TSS	Verify that the thermometer does not have mercury separation and annual calibration has occurred.	Perform as needed	178 – 182° C for TDS; 103 – 105° C for TSS	Repeat maintenance activity or remove from service		3.5.19.2 3.5.19.1

SAP Worksheet #26: Sample Handling System

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): Jason Oliver, Joe Burawa, Dave Gaviglia/Baker
Sample Packaging (Personnel/Organization): Jason Oliver/Baker
Coordination of Shipment (Personnel/Organization): Jason Oliver/Baker
Type of Shipment/Carrier: Priority Overnight/Federal Express

SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Cathy Dover/CompuChem
Sample Custody and Storage (Personnel/Organization): Cathy Dover/CompuChem
Sample Preparation (Personnel/Organization): Bradley Lake (organics); Joe Mohn (inorganics)/CompuChem
Sample Determinative Analysis (Personnel/Organization): Bradley Lake (organic GC/MS and GC analysis); Ron Harris (VOC GC/MS analysis); Joe Mohn (inorganics)/CompuChem

SAMPLE ARCHIVING
Field Sample Storage (No. of days from sample collection): 90 days
Sample Extract/Digestate Storage (No. of days from extraction/digestion): 13 months
Biological Sample Storage (No. of days from sample collection): NA

SAMPLE DISPOSAL
Personnel/Organization: Cathy Dover/CompuChem
Number of Days from Analysis: Approximately 60 days

SAP Worksheet #27: Sample Custody Requirements

An essential requirement of any sampling and analysis program is the ability to document sample history. Chain-of-custody establishes procedures and the documentation and control required to identify and trace a sample from collection to final analysis. These procedures include labeling to prevent sample misidentification, using custody seals to prevent unauthorized tampering with contents, securing custody, and maintaining the necessary records to support potential litigation and refute challenge of the data. The chain-of-custody history must document a legally acceptable record that covers all aspects of the sample collection, post-sampling handling, storage, and analytical process. This originates with the sample collection and indicates all custody transfers through final disposition of the sample. The custody procedures described below will be followed to ensure that the integrity of the samples are maintained throughout the course of the collection, handling, and analysis process.

27.1 Sample Nomenclature

The samples collected during the field program, including QA/QC samples, will be designated with a unique number. The number will serve to identify the SWMU, sample media or type, sampling location, depth, and QA/QC qualifier. The sample designation format will be as follows:

SWMU No. - Sample Media or Type/Location - Depth and/or QA/QC Qualifier

An explanation of each of these identifiers is provided below.

SWMU No.: This corresponds to SWMU 60 – Former Landfill at the Marina

Sample Media or Type:
SB – Soil Boring
GW – Groundwater
SD – Sediment
TB – Trip Blank
FB – Field Blank
ER – Equipment Rinsate Blank

Location: Each sample location will be identified with a unique identification number.

Depth: Depth indicators will be used for soil samples. The number will reference the depth interval of the sample. For example:

00 – Ground surface to 1 foot bgs
01 – 1 to 3 feet bgs
02 – 3 to 5 feet bgs

QA/QC:
D – Field duplicate sample (following depth)
MS/MSD – MS/MSD sample (following depth)

Under this sample designation format, the sample number 60-SB06-01D refers to:

60-SB06-01D	SWMU 60 – Former Landfill at the Marina
60-SB06-01D	Soil sample from a boring
60-SB06-01D	Soil boring 06
60-SB06-01D	Sample depth interval 1 to 3 feet bgs
60-SB06-01D	Field duplicate (QA/QC) sample

The sample number 60-GW05 refers to:

60-GW05	SWMU 60 – Former Landfill at the Marina
60-GW05	Groundwater sample from a monitoring well
60-GW05	Monitoring well SB05

This sample designation format will be followed throughout the field program. Required deviations to this format in response to field conditions will be documented in the field logbook(s).

27.2 Sample Container Labeling

Each sample will be adequately marked for identification from the time of collection through storage and shipping using standard sample container labels (Appendix F). Labels will not be affixed to container lids or caps. Indelible ink will be used to fill out the labels. In addition, clear tape will be placed over the labels (except on VOC and TPH GRO containers) to preserve the information. Clear tape will not be placed over the labels on VOC and TPH GRO containers for soil and groundwater samples because the tape can affect the final weights of soil samples and interfere with the robotics of auto-samplers for both soil and groundwater samples. At a minimum, the labels will include the following information:

- Project name
- Project number
- Sample identification number
- Date and time of sample collection
- Sampler name(s)
- Analysis requested
- Preservatives

27.3 Sample Preservation and Handling

Samples will be collected by a qualified field team member(s) under the supervision of the FOL. As samples are collected, they will be placed into appropriate, laboratory-supplied containers with the appropriate preservatives and labeled as described above. The specific sample containers, sample volumes, preservation requirements, and holding times are provided on Worksheet #19. The samples collected will be properly packaged in coolers and maintained at 4° C (+/- 2° C) during storage and shipment to the laboratory.

27.4 Custody Procedures

Chain-of-custody forms will be used to summarize and document pertinent sample information such as sample identification and type, matrix, date and time of collection, preservation, and requested analysis. Furthermore, the chain-of-custody forms will document sample custody and tracking through the sequential signatures of various sample custodians (e.g., sampler, air bill number, laboratory sample custodian). Once samples have been packaged and shipped, the chain-of-custody copy and air bill receipt will become part of the QA record. Custody procedures will apply to all environmental and associated field QA/QC samples obtained as part of the data collection system. Custody procedures for field and laboratory activities are described below.

The person undertaking the actual sampling in the field will be responsible for care and custody of the samples collected until they are properly transferred. A chain-of-custody form (Appendix F) will be completed for each sample shipment. The form will be filled out in a legible manner, using indelible ink

and will be signed and dated by the sampler. Pertinent notes, such as whether the sample was field filtered, or whether the sample is suspected to be high in contaminant concentration, will also be noted on the form.

The chain-of-custody form will accompany the associated samples. When transferring possession of the samples, the individual(s) relinquishing and receiving will sign, date, and note the time on the form. This documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. Commercial carriers (e.g., Federal Express) will not be required to sign off on the custody form as long as the custody form is sealed inside the sample cooler and the custody seals remain intact.

A custody seal (Appendix F) will be part of the chain-of-custody process and used to document sample integrity during shipping and prior to receipt by the laboratory (i.e., to ensure that the samples have not been tampered with after they have been collected in the field). The custody seal(s) will be signed and dated, placed across the front and back openings of each sample cooler, and covered with clear plastic tape.

Samples for laboratory analysis will be delivered to CompuChem in Cary, North Carolina via Federal Express (priority overnight to arrive before 10:00 a.m. the following day). Upon receipt at the laboratory, the Sample Custodian (or representative) will unpack the coolers, verify the contents with the incoming chain-of-custody form, sign and date the chain-of-custody form, and take the temperature using the temperature blank and/or infrared thermometer. In addition, the Sample Custodian will check the pH values for aqueous samples (except samples for VOC and TPH GRO analyses) requiring preservative to ensure that they are within the acceptable range. The pH of aqueous samples for VOC and TPH GRO analyses will be checked by the analyst after the analysis has been conducted so as to not compromise the samples. Any discrepancies, breakage, or preservation issues will be noted on the chain-of-custody form/sample receipt form and reported to the Laboratory Project Manager and Baker Data Management Specialist. The sample Custodian will be responsible for verification of all data entered onto the laboratory sample custody records. The Sample Custodian will then deliver the chain-of-custody form and any other paper work (e.g., temperature or pH QA notice) to the Project Manager for Laboratory Information Management System (LIMS) entry and client contact (if needed). Additional details regarding laboratory custody procedures are provided in CompuChem's Quality Manual (Appendix D). An example chain-of-custody checklist, receiving logs, QA notices, and internal chain-of-custody form are included in the Quality Manual.

SAP Worksheet #28.1: Laboratory QC Samples Table

Matrix: Soil, Groundwater, Solid IDW, Liquid IDW

Analytical Group: VOCs

Analytical Method/ SOP Reference: 8260B/1.3.2.2 and 1.3.2.4

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ except for common laboratory contaminants	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ except for common laboratory contaminants
Laboratory Control Sample (LCS)	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD Quality Systems Manual (QSM) 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs	Re-prepare and analyze all associated samples		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
MS	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
MSD	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs; the RPD should be ≤ 30%			Accuracy and Precision	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs; the RPD should be ≤ 30%
Internal Standard	Every field sample, standard, and QC sample	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or initial CCV on days when ICAL is not performed; EICP area within – 50% to +100% of ICAL midpoint	Inspect MS and GC for malfunctions; reanalysis of samples analyzed while system was malfunctioning is mandatory		Accuracy	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or initial CCV on days when ICAL is not performed; EICP area within – 50% to +100% of ICAL midpoint
Surrogate Spike	All field and QC samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits	For QC and field samples, correct problem then re-prepare and re-analyze all samples for failed surrogates in the associated preparatory batch; if obvious chromatographic interference is present, reanalysis may not be necessary; contact client for guidance		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits

SAP Worksheet #28.2: Laboratory QC Samples Table

Matrix: Soil, Groundwater, Sediment, Solid IDW, Liquid IDW

Analytical Group: LLPAHs

Analytical Method/ SOP Reference: 8270D-SIM/2.4.4.5

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs	Re-prepare and analyze all associated samples		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
MS	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
MSD	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs			Accuracy and Precision	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
Internal Standard	Every field sample, standard, and QC sample	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or initial CCV on days when ICAL is not performed; EICP area within – 50% to +100% of ICAL midpoint	Inspect MS and GC for malfunctions; reanalysis of samples analyzed while system was malfunctioning is mandatory		Accuracy	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or initial CCV on days when ICAL is not performed; EICP area within – 50% to +100% of ICAL midpoint
Surrogate Spike	All field and QC samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits	For QC and field samples, correct problem then re-prepare and re-analyze all samples for failed surrogates in the associated preparatory batch; if obvious chromatographic interference is present, reanalysis may not be necessary; contact client for guidance		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits

SAP Worksheet #28.3: Laboratory QC Samples Table

Matrix: Soil, Groundwater, Sediment, Solid IDW, Liquid IDW

Analytical Group: Pesticides

Analytical Method/ SOP Reference: 8081B/2.2.4.10

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs	Re-prepare and analyze all associated samples		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
MS	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs
MSD	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs; RPD ≤ 30%			Accuracy and Precision	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs and the RPD ≤ 30%
Confirmation of positive results	All positive results must be confirmed on second column or detector	Calibration and QC criteria same as for initial or primary column analysis; RPD between primary and second column results ≤ 40%	Apply J-flag if RPD > 40%; discuss in case narrative		Accuracy	Calibration and QC criteria same as for initial or primary column analysis; RPD between primary and second column results ≤ 40%
Surrogate Spike	All field and QC samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits	For QC and field samples, correct problem then re-prepare and re-analyze all samples for failed surrogates in the associated preparatory batch; if obvious chromatographic interference is present, reanalysis may not be necessary; contact client for guidance		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits

SAP Worksheet #28.4: Laboratory QC Samples Table

Matrix: Soil, Groundwater, Sediment, Solid IDW, Liquid IDW

Analytical Group: Metals

Analytical Method/ SOP Reference: 6010C/3.2.1.6; 6020A/3.2.1.9; 7470A/7471B/3.3.4

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs; if control limits are not listed in DoD QSM 4.2, then adhere to vendor's Certificate of Analysis (COA)	Re-prepare and analyze all associated samples		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 with allowances for marginal exceedance failures as noted in the associated SOPs; if control limits are not listed in DoD QSM 4.2, then adhere to vendor's COA
MS	One per batch of up to 20 samples	The percent recovery should be 80-120%	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely; Additional QC tests (e.g., dilution test and post digestion spike addition) will be used to evaluate matrix effects		Accuracy	The percent recovery should be 80-120%
MSD	One per batch of up to 20 samples	The percent recovery should be 80-120% and the RPD ≤ 20%			Accuracy and precision	The percent recovery should be 80-120% and the RPD ≤ 20%
Serial Dilution (does not apply to Mercury	One per preparation batch	The result must agree within ± 10% of the original sample result	Perform a Post Digestion Spike		Accuracy and precision	The result must agree within ± 10% of the original sample result
Post Digestion Spike(does not apply to Mercury)	One when serial dilution fails, MS/MSDs do not meet acceptance limits, or analyte concentration(s) in all samples < 50x LOD	The percent recovery should be 80-120%	Qualify result with appropriate flag		Accuracy and precision	The percent recovery should be 80-120%
Internal Standard (6020A only)	Added to all instrument QC and field Samples	The absolute response must not deviate more than 30-120% of the original response	Perform multiples of 5X dilutions on field samples until requirements are met		Accuracy	The absolute response must not deviate more than 30-120% of the original response

SAP Worksheet #28.5: Laboratory QC Samples Table

Matrix: Soil, Groundwater, Solid IDW, Liquid IDW

Analytical Group: TPH GRO

Analytical Method/ SOP Reference: 8015C/1.1.3.1 and 1.1.3.2

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recovery should be 75-125%	Re-prepare and analyze all associated samples		Accuracy	The percent recovery should be 75-125%
MS	One per batch of up to 20 samples	The percent recovery should be 75-125%	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	The percent recovery should be 75-125%
MSD	One per batch of up to 20 samples	The percent recovery should be 75-125% and the RPD ≤ 30%			Accuracy and Precision	The percent recovery should be 75-125% and the RPD ≤ 30%
Surrogate Spike	All field and QC samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits	For QC and field samples, correct problem then re-prepare and re-analyze all samples for failed surrogates in the associated preparatory batch; if obvious chromatographic interference is present, reanalysis may not be necessary; contact client for guidance		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits

SAP Worksheet #28.6: Laboratory QC Samples Table

Matrix: Soil, Groundwater, Solid IDW, Liquid IDW

Analytical Group: TPH DRO

Analytical Method/ SOP Reference: 8015C/2.2.3.7

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recovery should be 50-150%	Re-prepare and analyze all associated samples		Accuracy	The percent recovery should be 50-150%
MS	One per batch of up to 20 samples	The percent recovery should be 50-150%	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	The percent recovery should be 50-150%
MSD	One per batch of up to 20 samples	The percent recovery should be 50-150% and the RPD ≤ 30%			Accuracy and Precision	The percent recovery should be 50-150% and the RPD ≤ 30%
Surrogate Spike	All field and QC samples	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits	For QC and field samples, correct problem then re-prepare and re-analyze all samples for failed surrogates in the associated preparatory batch; if obvious chromatographic interference is present, reanalysis may not be necessary; contact client for guidance		Accuracy	The percent recoveries should meet control limits listed in DoD QSM 4.2 if available; otherwise use in-house control limits

SAP Worksheet #28.7: Laboratory QC Samples Table

Matrix: Groundwater

Analytical Group: TDS and TSS

Analytical Method/ SOP Reference: 2540C/3.5.19.2 and 2540D/3.5.19.1

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Accuracy	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recovery must be 90-110%	Re-prepare and analyze all associated samples		Accuracy	The percent recovery must be 90-110%
Duplicate	One per batch of up to 20 samples	The sample and duplicate must agree within 5% of their average weight	Contact client for guidance		Accuracy and Precision	The sample and duplicate must agree within 5% of their average weight

SAP Worksheet #28.8: Laboratory QC Samples Table

Matrix: Sediment

Analytical Group: Total Organic Carbon

Analytical Method/ SOP Reference: Lloyd Kahn/3.6.2.2

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	The percent recovery should be 90-110%	Re-prepare and analyze all associated samples		Accuracy	The percent recovery should be 90-110%
MS	One per batch of up to 20 samples	The percent recovery should be 75-125%	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	The percent recovery should be 75-125%
MSD	One per batch of up to 20 samples	The percent recovery should be 75-125% and the RPD ≤ 20%			Accuracy and Precision	The percent recovery should be 75-125% and the RPD ≤ 20%

SAP Worksheet #28.9: Laboratory QC Samples Table

Matrix: Solid IDW, Liquid IDW

Analytical Group: Ignitability

Analytical Method/ SOP Reference: ASTM D93-80/3.5.9.1

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
LCS	One per batch of up to 20 samples	Flash point must be 81° F ± 2° F	Re-analyze all associated samples	Analyst	Accuracy	Flash point must be 81° F ± 2° F
Duplicate	One per batch of up to 20 samples	Must agree within ± 2° F	Contact client for guidance		Accuracy and Precision	Must agree within ± 2° F

SAP Worksheet #28.10: Laboratory QC Samples Table

Matrix: Solid IDW, Liquid IDW

Analytical Group: Reactivity

Analytical Method/ SOP Reference: 9014/3.4.4 and 9034/3.5.18.1

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	All target analyte concentrations must be ≤½ LOQ	Re-prepare and analyze all associated samples	Analyst	Contamination and Bias	All target analyte concentrations must be ≤½ LOQ
LCS	One per batch of up to 20 samples	Recovery must be within 14-77% for cyanide; recovery must be within 50-150% for sulfide	Re-prepare and analyze all associated samples		Accuracy	Recovery must be within 14-77% for cyanide; recovery must be within 50-150% for sulfide
MS	One per batch of up to 20 samples	Recovery must be within 14-77% for cyanide; recovery must be within 50-150% for sulfide	If both the LCS and MS/MSD recoveries are unacceptable, re-prepare and analyze the associated samples and QC; if the LCS recovery is acceptable and the MS/MSD recoveries are unacceptable, matrix effect is likely and corrective action will not be taken		Accuracy	Recovery must be within 14-77% for cyanide; recovery must be within 50-150% for sulfide
MSD	One per batch of up to 20 samples	Recovery must be within 14-77% for cyanide; recovery must be within 50-150% for sulfide and RPD of ± 20%			Accuracy	Recovery must be within 14-77% for cyanide; recovery must be within 50-150% for sulfide and RPD of ± 20%

SAP Worksheet #28.11: Laboratory QC Samples Table

Matrix: Soil, Solid IDW, Liquid IDW

Analytical Group: Corrosivity (pH)

Analytical Method/ SOP Reference: 9045D/3.5.14.3; 9040C/3.5.14.2

QC Sample	Frequency & Number	Method/SOP QC Acceptance Limits	Corrective Action	Person(s) Responsible for Corrective Action	DQI	Measurement Performance Criteria
LCS	One per batch of up to 20 samples	Acceptable criteria is determined by vendor and listed on the COA	Recalibrate pH meter	Analyst	Accuracy	Acceptable criteria is determined by vendor and listed on the COA
Duplicate	One per batch of up to 20 samples	Must agree within ± 0.10 pH unit of true value	Recalibrate pH meter		Accuracy and Precision	Must agree within ± 0.10 pH unit of true value

SAP Worksheet #29: Project Documents and Records Table

Document	Where Maintained
SAP (including regulator comments and response to comments)	Electronic Portable Document File (PDF) copy will be maintained on Baker's network server; hardcopy original will be maintained in the project file; uploaded to the NAPR website; archived at project closeout
Sample Collection Records - Field Logbooks - Soil Boring and Monitoring Well Construction Records - Daily Meter Calibration Records - Groundwater Sampling Forms - Chain-of-Custody Forms - Site Photographs	Electronic PDF copies will be maintained on Baker's network server; hardcopies will be maintained in the project file; included in the Full RFI Report; archived at project closeout
- Air Bills - Pertinent E-mail Correspondence/Telephone Logs - Corrective Action Forms	Electronic PDF copies will be maintained on Baker's network server; hardcopies will be maintained in the project file; archived at project closeout
Geophysical Survey Report/Data	Electronic PDF copy will be maintained on Baker's network server; hardcopy will be maintained in the project file; included in the Full RFI Report; archived at project closeout
Sampling Location Survey Data	Electronic data will be maintained on Baker's network server and included in the Full RFI Report; archived at project completion
IDW Disposal Records (e.g., waste profiles, manifests)	Electronic PDF copies will be maintained on Baker's network server; hardcopies will be maintained in the project file; archived at project closeout
Laboratory Documents and Records - Sample Receipt, Chain-of-Custody, and Tracking Records - Standards Traceability Logs - Equipment Calibration Logs - Sample Prep Logs - Extraction/Clean-up Records - Run Logs - Equipment Maintenance, Testing, and Inspection Logs - Corrective Action Logs - Reported Field Sample Results - Reported Results for Standards, QC Checks, and QC Samples - Instrument Printouts (Raw Data) for Field Samples, Standards, QC Checks, and QC Samples - Data Package Completeness Checklists - Sample Disposal Records - Pertinent E-mail Correspondence/Telephone Logs	Maintained by the laboratory (CompuChem) and included in the Level IV data packages; electronic PDF copies of the data packages will be maintained on Baker's network server; electronic data will be uploaded to the project database and included in the Full RFI Report; archived at project closeout
Puerto Rico Chemist Certifications	Electronic PDF copy will be maintained on Baker's network server; hardcopy will be maintained in the project file; included in the Full RFI Report; archived at project closeout
Data Assessment Documents and Records - Data Validation Reports - Pertinent E-mail Correspondence/Telephone Logs - Corrective Action Forms	Electronic PDF copies will be maintained on Baker's network server; hardcopies will be maintained in the project file; data validation narrative summaries will be included in the Full RFI Report; archived at project closeout
Full RFI Report (including regulator comments and response to comments)	Electronic PDF copy will be maintained on Baker's network server; hardcopy original will be maintained in the project file; uploaded to the NAPR website; archived at project closeout

SAP Worksheet #30: Analytical Services Table

Analytical services for this project will be provided by CompuChem in Cary, North Carolina. CompuChem is a DoD ELAP-certified and Navy-approved laboratory. Complete Level IV data packages with sequentially numbered pages will be provided by the laboratory for each SDG within 28 calendar days from receipt of the samples. In general, the Level IV data packages will include, but not be limited to, the following:

- Cover page (with laboratory name, address, phone number, contact person, SDG number, project name, and project number)
- Table of contents referencing individual sections
- Case narrative
- Sample management records including the original copy of chain-of-custody forms (including cooler temperature and sample condition), shipping documents, and laboratory sample receipt forms
- Cross-reference table of field sample identification to laboratory sample identification
- Analytical results and QA/QC information for organics analysis as follows:
  - Sample result forms (dry weight for solids) including method blanks
  - Percent solids calculations
  - Sample raw data
  - Surrogate summaries (surrogate results may appear on the sample result forms)
  - QC summaries
  - Tune data (GC/MS only)
  - ICAL with retention time information (When manual integration is performed, raw data recorded will include a complete audit trail for those manipulations, raw data output showing the results of the manual integration, and notation of rationale, date, and initials of person performing the manual integration)
  - Daily calibration checks including related CCVs
  - Resolution check standards (GC/MS and pesticides) (if applicable)
  - QC (LCS, MS/MSD) raw data
  - Instrument run logs
  - Sample preparation logs
- Analytical results and QA/QC information for inorganics analysis as follows:
  - Sample result forms (dry weight for solids) including method blanks
  - Percent solids calculations
  - Sample raw data
  - QC summaries
  - ICAL
  - Daily calibration checks including related CCVs
  - Calibration blanks including related continuing calibration blanks
  - Interference check standards A and B for ICP-AES only
  - QC raw data
  - Post-digestion spike results
  - Analytical spike results
  - Method of standard additions
  - ICP-AES serial dilutions
  - Instrument run logs
  - Sample preparation logs

Analytical results will also be provided in an EDD compatible with Windows applications. Specific laboratory EDD requirements are included in Appendix G.

SAP Worksheet #31: Planned Project Assessments

External audits may be conducted by the USEPA or PREQB at any time during the life cycle of the project. If an audit is to be conducted, scheduling should be coordinated to ensure that personnel and equipment are available as necessary.

One field sampling technical systems audit will be conducted at the start of field sampling activities so that effective corrective action measures can be implemented if warranted. The audit will be conducted by Baker's Activity Coordinator/Project Manager or appropriate designee. Throughout the field program, Baker's FOL will also monitor field personnel to ensure that sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures, are being conducted in accordance with this SAP. The FOL will be responsible for responding to any audit findings and implementing any required corrective action in a timely manner. Baker's Activity Coordinator/Project Manager, Task Manager, QA Officer, and FOL will be responsible for monitoring the effectiveness of any required corrective action implemented.

No laboratory assessments are anticipated for this project since CompuChem is a DoD ELAP-certified and Navy-approved laboratory. CompuChem's QA/QC Officer or appropriate designee performs routine internal audits of the laboratory. Data audits also are performed by the laboratory's QA Manager or designee on at least 10% of the QSM 4.2 DoD data packages. The data packages subject to audit are randomly selected by the Laboratory's QA Manager. In addition, each laboratory department at CompuChem analyzes blind PT samples. These internal systems audits are conducted to detect any problems in sample flow, analytical procedures, or documentation and to ensure adherence to the laboratory's SOPs.

SAP Worksheet #32: Assessment Findings and Corrective Action Responses

As indicated on Worksheet #31, one field sampling technical systems audit will be conducted at the start of field sampling activities. In addition, under the QA/QC program for this project, it is required that any and all personnel noting conditions adverse to quality report these conditions immediately to Baker's Activity Coordinator/Project Manager, Task Manager, and QA Officer. These parties, in turn along with the FOL, are charged with implementing appropriate corrective action in a timely manner. In addition, Baker's Activity Coordinator/Project Manager will notify the Navy, USEPA, and PREQB of any significant corrective actions implemented. It is ultimately the responsibility of these personnel to monitor the effectiveness of the corrective measures performed. Any corrective actions regarding sample collection, handling, and shipping protocols, as well as equipment decontamination and field documentation procedures will be immediately addressed in the field.

SAP Worksheet #33: Quality Assurance Management Reports Table

Type of Report	Frequency (daily, weekly monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (title and organizational affiliation)	Report Recipient(s) (title and organizational affiliation)
Field Progress Reports	Daily	Every day that field sampling and data collection activities are occurring	Jason Oliver/FOL/Baker	Mark Kimes/Project Manager/Baker; Rick Aschenbrenner/QA Officer/Baker; Scott Moffett/Task Manager/Baker; Shannon Raub/Data Management Specialist/Baker
Data Validation Reports	Once (for each SDG)	14 days after receipt of final, complete Level IV analytical data package	Laura Maschhoff and Jackie Cleveland/Data Validator/DataQual	Mark Kimes/Project Manager/Baker
Full RFI Report	Once	60 days after receipt of validated analytical data	Scott Moffett/Task Manager/Baker	Stakeholders (see Worksheet #3)
RCRA Progress Reports	Quarterly	Quarterly during duration of the project	Mark Kimes/Project Manager/Baker	Stakeholders (see Worksheet #3)

Note:
The specific content of each QA management report is provided on Worksheet #14.

SAP Worksheet #34: Data Verification (Step I) Process Table

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Planning Documents	Ensure approval and completeness of SAP	Internal	Mark Kimes (Project Manager)/Baker
Field Measurements	Field personnel will ensure that the equipment used is performing accurately (see Worksheet #22)	Internal	Jason Oliver (FOL)/Baker
Chain-of-Custody Forms and Shipping Documentation	Chain-of-custody forms and shipping documentation will be reviewed internally upon their completion and verified against the packed sample coolers they represent. Once everything checks out, the chain-of-custody forms will be signed. One copy of the chain-of-custody forms will be retained in the site file, and the original and remaining copies will be placed inside the cooler for shipment.	Internal	Jason Oliver (FOL)/Baker
Field Logbooks	Field logbooks will be reviewed to ensure completeness such that site activities can be reconstructed. The logbooks will also be used to document, explain, and justify all deviations from the approved SAP.	Internal	Scott Moffett (Task Manager)/Baker
Field Data Collection Forms (e.g., Daily Meter Calibration Records, Groundwater Sampling Forms, Soil Boring and Well Construction Records)	Field Data Collection Forms will be reviewed to ensure completeness of field data, sample collection times, etc.	Internal	Scott Moffett (Task Manager)/Baker
Field Investigation Interpretive Data (e.g., Geophysical Survey Data, Sampling Location Survey Data, Slug Test Data)	Upon receipt, the data will be reviewed to ensure completeness and accuracy of the data collected.	Internal	Scott Moffett (Task Manager)/Baker
Daily Field Sample Summaries	Daily field sample summaries will be reviewed upon receipt each day to ensure the samples identified in this SAP were collected. The summaries will also be used for sample tracking.	Internal	Scott Moffett (Task Manager)/Baker
Sample Login/Receipt	Upon receipt at the laboratory, the samples will be unpacked and cross-referenced against the chain-of-custody forms. Any discrepancies and/or broken containers will be communicated to the Baker Data Management Specialist. Sample log-in/receipt forms, including sample identifications and requested analyses, will be submitted to the Baker Data Management Specialist.	Internal	Cathy Dover (Laboratory Project Manager)/CompuChem
	Upon receipt, the sample log-in/receipt forms will be reviewed to ensure the samples received at the laboratory are scheduled to be analyzed for the correct parameters specified in this SAP.	Internal	Shannon Raub (Data Management Specialist)/Baker

SAP Worksheet #34: Data Verification (Step I) Process Table (*continued*)

Verification Input	Description	Internal/ External	Responsible for Verification (name, organization)
Laboratory Analytical Data Packages and EDDs (see Worksheet #30 for specific content)	Laboratory data packages and EDDs will be reviewed to ensure completeness and technical accuracy prior to submittal to Baker.	Internal	Cathy Dover (Laboratory Project Manager)/CompuChem
	Prior to submittal to the data validator, laboratory data packages and EDDs will be reviewed to ensure the samples identified in this SAP were analyzed by the laboratory for the correct parameters and all data is present.	External	Shannon Raub (Data Management Specialist)/Baker
	Upon receipt, laboratory data packages and EDDs will be reviewed to ensure completeness. Missing information will be requested from the laboratory, and validation will be suspended until missing data are received.	External	Laura Maschhoff and Jackie Cleveland (Data Validators)/DataQual
Data Validation Reports (see Worksheet #14 for specific content)	Data validation reports will be reviewed for completeness. In addition, the hardcopy data (e.g., Form 1's) will be compared to the EDD (with validator qualifiers incorporated) to ensure accuracy.	External	Shannon Raub (Data Management Specialist)/Baker

SAP Worksheet #35: Data Validation (Steps IIa and IIb) Process Table

Step IIa/IIb	Validation Input	Description	Responsible for Validation (name, organization)
Step IIa	SAP	Determine whether this SAP was executed as specified (i.e., the number, location, and type of field samples were collected and analyzed).	Scott Moffett (Task Manager)/Baker
	Field Procedures and Sampling Methods	Review field logbooks to ensure that field procedures/sampling methods were followed (see Worksheet #14) and any deviations were documented.	
	Analytical Methods/SOPs	Review laboratory case narratives and analytical data packages to ensure that analytical methods/SOPs were followed (see Worksheet #23).	Laura Maschhoff and Jackie Cleveland (Data Validators)/DataQual
	Analytes	Review laboratory analytical data packages to ensure the required lists of analytes were reported as specified in this SAP.	
	Holding Times	Review laboratory analytical data packages to ensure the samples were analyzed within holding times specified in this SAP. If holding times were not met, confirm that deviations were documented, that appropriate notifications were made, and that approval to proceed was received prior to analysis.	
Step IIb	SAP Deviations	Determine the impacts of any deviations from sampling or analytical methods and SOPs (i.e., does the data still meet measurement performance criteria). Consider the effectiveness and appropriateness of any corrective action.	Scott Moffett (Task Manager)/Baker; Laura Maschhoff and Jackie Cleveland (Data Validators)/DataQual
	Field Data	Review field logbooks and field data collection forms to ensure that field data meet SAP requirements for completeness and accuracy based on field calibration records.	Scott Moffett (Task Manager)/Baker
	Project QLs	Ensure sample results met the project quantitation and action limits specified on Worksheet #15.	Shannon Raub (Data Management Specialist)/Baker
	QA/QC Results	Review laboratory analytical data packages to ensure that field and laboratory QA/QC samples were run and compliant with method-required limits as specified on Worksheets #12 and #28.	Shannon Raub (Data Management Specialist)/Baker; Laura Maschhoff and Jackie Cleveland (Data Validators)/DataQual
	Raw Laboratory Data	Review 10% of the data to confirm calibrations and quantitation of reported results. Raw laboratory data for this review will be randomly selected by the validator.	Laura Maschhoff and Jackie Cleveland (Data Validators)/DataQual

Notes:
IIa – Compliance with methods, procedures, and contracts
IIb – Comparison with measurement performance criteria in the SAP

SAP Worksheet #36: Analytical Data Validation (Steps IIa and IIb) Summary Table

Step IIa/IIb	Matrix	Analytical Group	Validation Criteria	Validator (name, organization)
Step IIa	Soil	Appendix IX VOCs, LLPAHs, Pesticides, Metals, TPH GRO and DRO	The analytical data will be validated in accordance with the SW-846 methods utilized by the laboratory, specifications set forth in the most recent USEPA Region II SOPs for Validation of Organic Data Acquired using SW-846 Methods, and professional judgment. Region II has not developed validation checklist SOPs for the methods used to assess inorganics and TPH GRO/DRO. Therefore, alternative worksheets will be used (see Appendix E). Non-compliant analytical results will be qualified using Region II flagging conventions.	Laura Maschhoff and Jackie Cleveland/DataQual
	Groundwater	Appendix IX VOCs, LLPAHs, Pesticides, Metals (total and dissolved fractions), TPH GRO and DRO, TDS, TSS		
	Sediment	Appendix IX LLPAHs, Pesticides, Metals, TOC		
Step IIb	Soil	Appendix IX VOCs, LLPAHs, Pesticides, Metals, TPH GRO and DRO	The analytical data will be compared to the PALs provided on Worksheet #15, ecological and human health, risk-based screening values included in Appendix A, and background values for metals (ULM) (Baker, 2010).	Shannon Raub/Baker
	Groundwater	Appendix IX VOCs, LLPAHs, Pesticides, Metals (total and dissolved fractions), TPH GRO and DRO, TDS, TSS		
	Sediment	Appendix IX LLPAHs, Pesticides, Metals, TOC		

Notes:
IIa – Compliance with methods, procedures, and contracts
IIb – Comparison with measurement performance criteria in the SAP

SAP Worksheet #37: Usability Assessment

The data usability assessment is an evaluation based on the results of data verification and validation in the context of overall project decisions or objectives. The assessment determines whether the project execution and resulting data meet the PQOs. Both the sampling and analytical activities must be considered with the ultimate goal of assessing whether the final, qualified results support the decisions to be made with the data.

The following sections summarize the processes that will be used to determine whether the data collected are of the right type, quality, and quantity to support environmental decision making for the project. In addition, the following sections describe how data quality issues will be addressed, and how limitations of the use of the data will be handled.

37.1 Usability Assessment Process and Procedures

Upon completion of the field program and receipt/validation of the analytical data, the project team will meet to initiate the usability assessment process and ensure that the PQOs are understood and the full scope is considered. Activities that will be considered during the usability assessment are summarized below.

Considerations for Usability Assessment

Item	Assessment Activity	Responsible Person (name, organization)
SAP and Data Deliverables	The SAP and data deliverables will be reviewed to ensure that all necessary information was provided, including but not limited to validation results.	Scott Moffett and Shannon Raub/Baker
Deviations	Deviations will be evaluated to determine any impact on the usability of data.	Scott Moffett/Baker; Laura Maschhoff and Jackie Cleveland/ DataQual
Sampling Locations	Sampling locations adjusted in the field will be reviewed to determine if the locations continue to satisfy the project objectives.	Scott Moffett/Baker
Chain-of-Custody	Chain-of-custody forms will be reviewed to establish that any problems with documentation or custody procedures do not prevent the data from being used for the intended purpose.	Scott Moffett and Shannon Raub/Baker
Holding Times	Analytical data will be reviewed to determine the acceptability of data where holding times were exceeded.	Laura Maschhoff and Jackie Cleveland/DataQual
Damaged Samples	Analytical data will be reviewed to determine whether data from damaged samples are usable. If the data cannot be used, a determination will be made as to whether re-sampling is necessary.	Laura Maschhoff and Jackie Cleveland/DataQual
SOPs and Methods	SOPs and methods will be reviewed to evaluate the impact of deviations on data quality.	Scott Moffett/Baker; Laura Maschhoff and Jackie Cleveland/ DataQual
QC Samples	Analytical data will be reviewed to evaluate the implications of unacceptable QC sample results on the data usability for associated samples.	Laura Maschhoff and Jackie Cleveland/DataQual
Matrix	Analytical data will be evaluated to determine matrix effects (interference or bias).	Laura Maschhoff and Jackie Cleveland/DataQual
Meteorological Data and Site Conditions	Data will be evaluated to determine the possible effects of meteorological (e.g. wind, rain, temperature) and site conditions on sample results. Any unusual conditions that were present at the time of sampling and data collection will be identified.	Scott Moffett/Baker
Comparability	Sampling and analysis methods and data reporting formats will be reviewed to ensure results from different data collection activities achieve an acceptable level of agreement.	Scott Moffett/Baker
Completeness	The impact of missing information will be evaluated to ensure that enough information was obtained for the data to be usable.	Scott Moffett/Baker; Laura Maschhoff and Jackie Cleveland/ DataQual
Critical Samples	Data will be evaluated to establish that critical samples and critical target analytes, as defined in this SAP, were collected and analyzed.	Scott Moffett/Baker
Data Restrictions	Analytical results that do not comply with the data validation guidelines (see Worksheet #36) will be qualified with an associated explanatory note. In most cases, these results will represent minor quality control problems (i.e., typical analytical difficulties or the result of sample matrix issues) and will not affect data usability. Data rejected (qualified as R) as a result of the validation process will be treated as unreliable and unusable and will not be used for environmental decision-making.	Laura Maschhoff and Jackie Cleveland/DataQual

Specifically, the data usability assessment process will consist of the following steps:

- Step 1 – Data usability assessment focused on the quality of the data generated as compared to the PQOs and measurement performance criteria of precision, accuracy/bias, and sensitivity and the use or limitations of such data for project decisions.

- Step 2 – Data usability in terms of the usefulness of the project data as a whole in making project decisions and overall evaluation of representativeness, completeness, and comparability.

- Reconciliation with the PQOs

37.1.1 Step 1 – Data Usability in Terms of Precision, Accuracy/Bias, and Sensitivity

The first step of the data usability assessment will be performed primarily by DataQual during the data validation process. Specific restrictions on data, such as data that would be qualified as estimated (J), will be determined and documented in the data validation reports. If cumulative QC exceedances are noted that cause severe uncertainty in a result(s), these data will be considered unusable for project decisions.

The following measurement performance criteria will be assessed and conclusions drawn on usability of the data based on their results as the first step of the data usability assessment process.

Precision

Precision is the degree to which a set of observations or measurements of the same matrix, obtained under similar conditions, conform to themselves. Field duplicate samples and MSD samples will be analyzed at the frequency described on Worksheets #12 and #14 to assess field and laboratory precision. Overall precision will be measured during the data validation process by the RPD (i.e., variability) between each sample (or spike) and the corresponding duplicate (or duplicate spike). The following equation will be used to calculate the RPD:

$$RPD = \frac{X_1 - X_2}{(X_1 + X_2)/2} \times 100\%$$

Where:

X_1 = original sample concentration
X_2 = duplicate sample concentration

These results will be checked against the measurement performance criteria for each respective matrix and analytical group presented on Worksheet #12. Results for analytes that exceed the criteria will be identified in the data validation reports. Appropriate actions will be taken to qualify the associated results, as necessary, based on validation guidance presented on Worksheet #36. Validation actions in response to the precision evaluation will be summarized in the data validation reports and included in the Full RFI report. In addition, conclusions and any limitations on use of the data will be presented in the Full RFI report.

Accuracy/Bias

Accuracy is the degree of agreement between an observed value (sample result) and an accepted reference value; bias describes the systematic or persistent distortion associated with a measurement process. The

terms "accuracy" and "bias" are used interchangeably herein. Accuracy in the field will be assessed through the use of trip blanks, field blanks, and equipment rinsate blanks (see Worksheets #12 and #14) and will be ensured through adherence to all sample handling, preservation, and holding time requirements (see Worksheets #14 and #19).

Accuracy in the laboratory will be assessed through the use of laboratory method blanks and instrument blanks. In addition, accuracy in the laboratory will be assessed through comparison of a spiked sample result with a known or calculated value and is expressed as a Percent Recovery (%R). Percent recoveries will be derived from analysis of known amounts of compounds spiked into laboratory-grade DI water (i.e., LCS analysis) or into actual samples (i.e., surrogate or MS analyses). LCS analysis, which may also be referred to as blank spike analysis, measures the accuracy of laboratory operations. Surrogate and MS analyses measure the accuracy of laboratory operations as affected by sample matrix. LCS, surrogate, and MS analyses will be performed at a frequency described on Worksheet #28. Laboratory accuracy will be assessed via comparison of the calculated %R values with measurement performance criteria specified on Worksheet #28. The %R for a spiked sample will be calculated by using the following equation:

$$\%R = \frac{\textit{Spiked Sample Concentration} - \textit{Unspiked Sample Concentration}}{\textit{Spiked Concentration Added}} \; x\ 100\%$$

The %R for LCS and surrogate compound results will be calculated using the following equation:

$$\%R = \frac{\textit{Experimental Concentration}}{\textit{Known Concentration Added}} \; x\ 100\%$$

Results for analytes that exceed the criteria will be identified in the data validation reports. Appropriate actions will be taken to qualify associated results, as necessary, based on validation guidance presented on Worksheet #36. Validation actions in response to the accuracy/bias evaluation will be summarized in the data validation reports and included in the Full RFI report. In addition, conclusions and any limitations on use of the data will be presented in the Full RFI report.

Sensitivity

Results for low-level calibration checks and calibration criteria will be evaluated during the data validation process. These results will be checked against the measurement performance criteria presented on Worksheet #24. Appropriate actions will be taken to qualify associated results, as necessary, based on validation guidance presented on Worksheet #36. Validation actions in response to the sensitivity evaluation will be summarized in the data validation reports and included in the Full RFI report.

Sensitivity will also be evaluated by the Baker Data Management Specialist (Shannon Raub) based on comparison of the sample-specific LODs for non-detect results to the ecological and human health, risk-based screening values summarized in Appendix A. Non-detect results that show elevated quantitation limits above the screening values will be identified and evaluated for usability with respect to meeting PQOs. Findings of the sensitivity evaluation and any limitations on use of the data will be presented in the Full RFI report.

37.1.2 Step 2 – Data Usability in Terms of Representativeness, Completeness, and Comparability

The second step of the data usability assessment concerning achievement of the overall PQOs in a holistic approach will be conducted by the Baker Task Manager (Scott Moffett) with assistance from the Data Management Specialist (Shannon Raub) for queries from the project database. This will involve a review

of chemical and non-chemical information generated during the field program to evaluate representativeness, completeness, and comparability of the data as discussed below.

Representativeness

Representativeness is a qualitative term that describes the extent to which the sampling design adequately reflects the environmental conditions of a site. Representativeness also reflects the ability of the sampling team to collect samples and the ability of laboratory personnel to analyze those samples so that the generated data accurately and precisely reflect site conditions. Use of standardized sampling, handling, analytical, and reporting procedures ensures that the final data accurately represent actual site conditions.

Representativeness in this sampling and analysis program will be ensured by adhering to the SAP and using proper sampling techniques. From the analytical end, representativeness will be ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing field duplicate samples. The sampling network was designed to provide data representative of conditions at the SWMU. Decisions regarding the sampling design and rationale are documented on Worksheets #10, #11, and #17.

The representativeness evaluation will be accomplished by verifying that the samples were collected and processed/analyzed in accordance with this SAP, by reviewing spatial and/or temporal data variations, and by comparing these characteristics to expectations. This will not require quantitative comparisons unless professional judgment indicates that a quantitative analysis is warranted. Findings of the representativeness evaluation and any limitations on use of the data will be presented in the Full RFI report.

Comparability

Comparability is a qualitative indicator of the confidence with which one data set can be compared to another (e.g., between sampling points, between sampling events). Comparability will be achieved by using standardized sampling and analysis methods and data reporting formats (including use of consistent units of measure). Complete field documentation using standardized data collection forms will support the assessment of comparability. The matrices sampled and range of field conditions encountered will also be considered in determining comparability.

Assessment of comparability is primarily subjective and will be accomplished by verifying adherence to the field procedures and analytical SOPs specified in this SAP (see Worksheets #14 and #23). Findings of the comparability evaluation and any limitations on use of the data will be presented in the Full RFI report.

Completeness

Completeness is a measure of the amount of valid data collected using a measurement system. It is expressed as a percentage of the number of measurements expected to be collected under correct, normal conditions. Completeness is calculated for the aggregation of data for each analyte measured for any particular sampling event or other defined set of samples (i.e., by SWMU) as specified in the PQOs.

The ideal objective for completeness is 100% (i.e., every sample planned to be collected is collected; every sample submitted for analysis yields valid data). However, samples can be rendered unusable during shipping or preparation (e.g., bottles broken or extracts accidentally destroyed), errors can be introduced during analysis (e.g., loss of instrument sensitivity, introduction of ambient laboratory

contamination), or strong matrix effects can become apparent (e.g., extremely low MS recovery). These instances result in data that do not meet QC criteria.

All of the data (except for solid and liquid IDW data) will be subjected to independent, third-party data validation, which will be conducted by DataQual of St. Louis, Missouri (see Worksheets #14 and #36). Data rejected (qualified as R) as a result of the validation process will be treated as unreliable and unusable. Upon completion of the validation process, completeness will be calculated for each method, matrix, and analyte combination using the following equation and summarized in the Full RFI report.

$$\%C = \frac{V}{T} \text{ x } 100\%$$

Where:

$\%C$ = percent completeness
V = number of valid measurements
T = total number of measurements

For project completeness, 90 to 95% of all sample data for a given analyte must represent valid measurements (Baker, 1995). Any analyte group that does not achieve the completeness goal may be considered a potential data gap. The need for further investigation and/or re-sampling will be determined on a case-by-case basis depending on whether data can be extrapolated from adjacent sample locations and whether the data are needed based on the results from adjacent sample locations.

37.1.3 Reconciliation with the PQOs

The final step of the data usability assessment is to evaluate whether the data meet the planned PQOs for the project. The final results, as adjusted for the findings of any data validation and data evaluation, will be checked against the PQOs, and an assessment will be made as to whether the data are of sufficient quality to support the PQOs. The decision as to data sufficiency may be affected by the overall precision, accuracy, and completeness of the data as demonstrated by the data validation process. The main project objective should be met assuming the 90% completeness goal is obtained after all of the data have undergone sufficient data validation. If significant deficiencies affecting achievement of the PQOs are identified, the deficiencies will be reviewed in a face-to-face meeting or teleconference with the Navy, USEPA, and PREQB. If no significant deficiencies are identified, the data usability assessment will simply be documented in the Full RFI report and reviewed during the normal document review cycle.

In addition to the assessments described above, statistical evaluations may be conducted. The statistical evaluations will be conducted by the Baker Task Manager (Scott Moffett) with assistance from the Data Management Specialist (Shannon Raub) and include simple summary statistics for target analytes such as minimum detected concentration, maximum detected concentration, number of non-detect results, number of detected results, frequency of detection, and location of maximum detected result. For statistical comparisons, non-detect results will be represented by a concentration equal to the analyte-specific LOD. For duplicate sample results, the most conservative value will be used for project decisions.

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FIGURES

K:_SOUTHNAVFAC\125201 JM07\SWMU 60\cad\125201_FULL_RFI_60_01.dwg

4 0 2 4 8

1 inch = 4 miles

Baker

FIGURE 1
NAPR LOCATION MAP
SWMU 60–FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

SOURCE: METRODATA, INC., 1999.

NAVAL ACTIVITY PUERTO RICO

K:_SOUTHNAVFAC\125201 JM07\SWMU 60\cad\125201_FULL_RFI_60_02.dwg

LEGEND

1 – SWMUs

60 – AREA TO WHICH THIS REPORT PERTAINS

AOC D – AOCs

SOURCE: GEO-MARINE, INC., SEPTEMBER 6, 2000.

3000 0 1500 3000

1 inch = 3000 ft.

FIGURE 2
SWMU/AOC LOCATION MAP
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO

IMAGE SOURCE: USGS 2006.

120 0 60 120
1 inch = 120 ft.

Baker

LEGEND

- SWMU BOUNDARY
- SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
- ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
- GASOLINE/DIESEL ASTs
- APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
- OUTFALL

COWARDIAN WETLAND CLASSIFICATION

E2SS3 - ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.

M2US2 - MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

FIGURE 3
SITE LAYOUT
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO

K:_SOUTHNAVFAC\125201 JM07\SWMU 60\cad\125201_FULL_RFI_60_04.dwg

UPLAND
UPLAND
UPLAND
UPLAND
UPLAND
UPLAND
E2SS3
E2SS3
E2SS3
M2US2

FORMER ATHLETIC FIELDS

TOW WAY DRIVE

60SD03
60SD02
60SD01

SB-12/MW-4
SB-11
SB-10
SB-6
60SB05
SB-3
SB-1/MW-1
SB-5/MW-3
SB-7
SB-4
60SB04
SB-8
SB-9/MW-2
SB-2
6E-SW/SD01
6E-01
60SB03
6E-02
60SB02
60SB01
6E-03
6E-SW/SD02

GASOLINE/DIESEL ASTs

ENSENADA HONDA

NOTES:
1. BORING/MONITORING WELL LOCATIONS FROM THE 1999 SITE CHARACTERIZATION ARE APPROXIMATE.
2. WELLS MW-2 AND MW-3 COULD NOT BE LOCATED DURING THE PHASE I RFI.

COWARDIAN WETLAND CLASSIFICATION

E2SS3 -ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.
M2US2 -MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

120 0 60 120
1 inch = 120 ft.

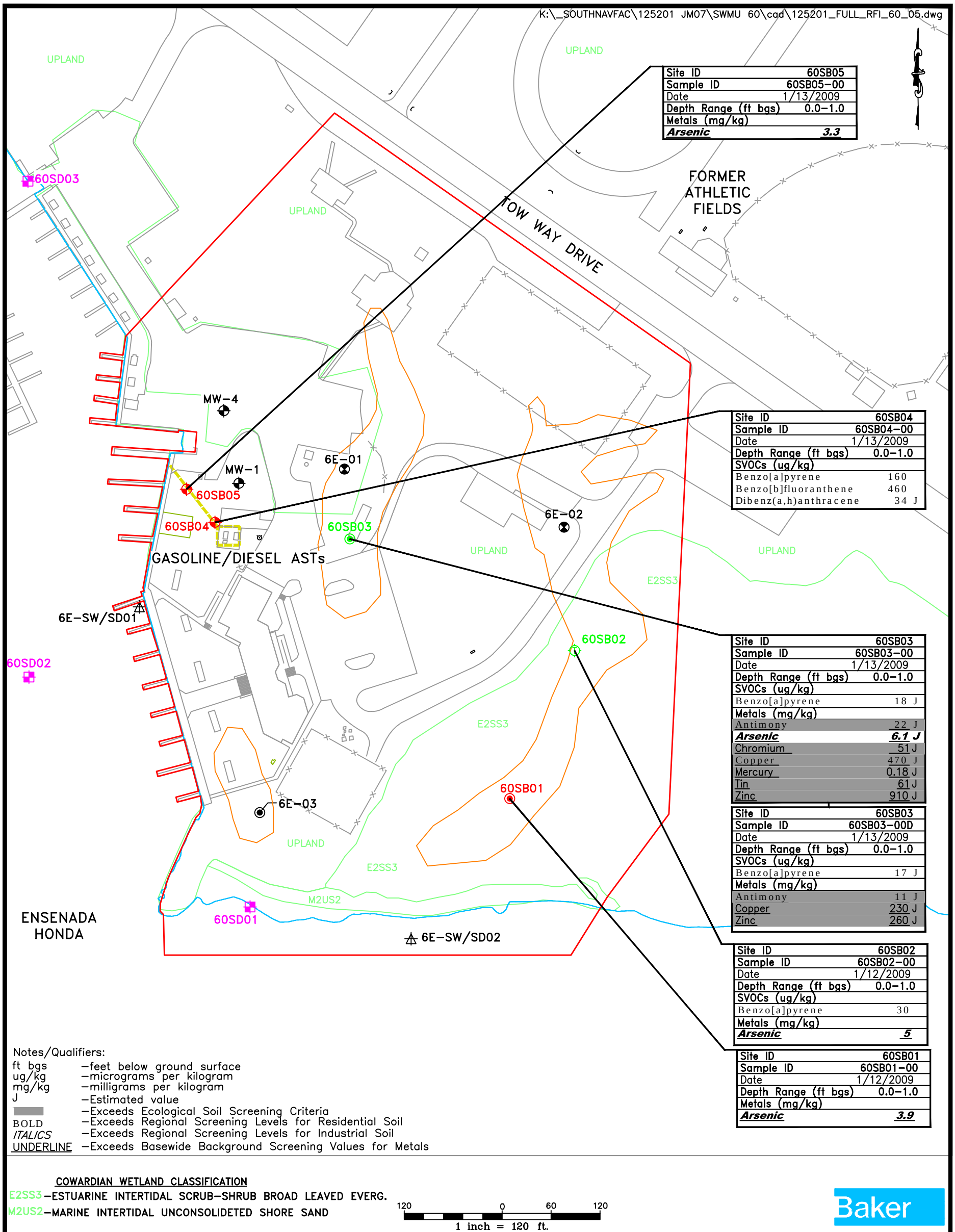
Baker

LEGEND

-SWMU BOUNDARY
-ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
-SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
-GASOLINE/DIESEL ASTs
-APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
-SOIL SAMPLE LOCATION (1999 SITE CHARACTERIZATION)
-SOIL AND GROUNDWATER SAMPLE LOCATION (1999 SITE CHARACTERIZATION)
-SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE II ECP 2004)
-SUBSURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (PHASE II ECP 2004)
-SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
-SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
-SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
-SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION (PHASE I RFI 2009)
-SURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (TEMPORARY) (PHASE I RFI 2009)
-SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)

FIGURE 4
HISTORICAL SAMPLE LOCATION MAP
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO



Site ID	60SB05
Sample ID	60SB05-00
Date	1/13/2009
Depth Range (ft bgs)	0.0-1.0
Metals (mg/kg)	
Arsenic	***3.3***

Site ID	60SB04
Sample ID	60SB04-00
Date	1/13/2009
Depth Range (ft bgs)	0.0-1.0
SVOCs (ug/kg)	
Benzo[a]pyrene	160
Benzo[b]fluoranthene	460
Dibenz(a,h)anthracene	34 J

Site ID	60SB03
Sample ID	60SB03-00
Date	1/13/2009
Depth Range (ft bgs)	0.0-1.0
SVOCs (ug/kg)	
Benzo[a]pyrene	18 J
Metals (mg/kg)	
Antimony	22 J
Arsenic	***6.1 J***
Chromium	51 J
Copper	470 J
Mercury	0.18 J
Tin	61 J
Zinc	910 J

Site ID	60SB03
Sample ID	60SB03-00D
Date	1/13/2009
Depth Range (ft bgs)	0.0-1.0
SVOCs (ug/kg)	
Benzo[a]pyrene	17 J
Metals (mg/kg)	
Antimony	11 J
Copper	230 J
Zinc	260 J

Site ID	60SB02
Sample ID	60SB02-00
Date	1/12/2009
Depth Range (ft bgs)	0.0-1.0
SVOCs (ug/kg)	
Benzo[a]pyrene	30
Metals (mg/kg)	
Arsenic	***5***

Site ID	60SB01
Sample ID	60SB01-00
Date	1/12/2009
Depth Range (ft bgs)	0.0-1.0
Metals (mg/kg)	
Arsenic	***3.9***

Notes/Qualifiers:

ft bgs	-feet below ground surface
ug/kg	-micrograms per kilogram
mg/kg	-milligrams per kilogram
J	-Estimated value
	-Exceeds Ecological Soil Screening Criteria
BOLD	-Exceeds Regional Screening Levels for Residential Soil
ITALICS	-Exceeds Regional Screening Levels for Industrial Soil
UNDERLINE	-Exceeds Basewide Background Screening Values for Metals

COWARDIAN WETLAND CLASSIFICATION

E2SS3 -ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.

M2US2 -MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

120 0 60 120

1 inch = 120 ft.

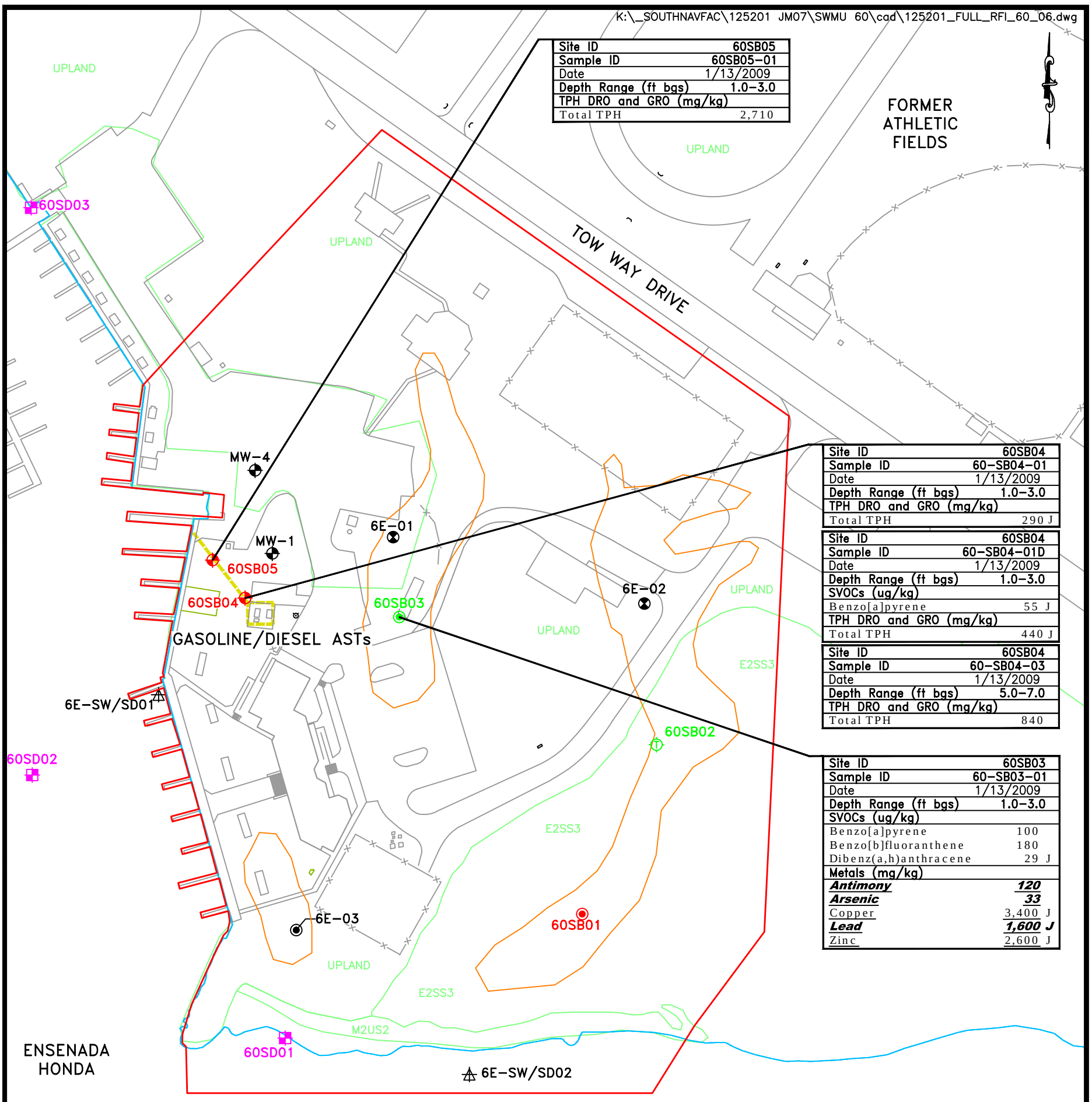
Baker

LEGEND

- -SWMU BOUNDARY
- -ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
- -SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
- -GASOLINE/DIESEL ASTs
- -APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
- -EXISTING MONITORING WELL LOCATION (1999 SITE CHARACTERIZATION)
- -SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE II ECP 2004)
- -SUBSURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (PHASE II ECP 2004)
- -SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
- -SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
- -SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
- -SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION (PHASE I RFI 2009)
- -SURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (TEMPORARY) (PHASE I RFI 2009)
- -SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)

FIGURE 5
SURFACE SOIL EXCEEDANCES OF HUMAN HEALTH, ECOLOGICAL, AND BACKGROUND SCREENING CRITERIA-PHASE I RFI
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO



Site ID	60SB05
Sample ID	60SB05-01
Date	1/13/2009
Depth Range (ft bgs)	1.0-3.0
TPH DRO and GRO (mg/kg)	
Total TPH	2,710

Site ID	60SB04
Sample ID	60-SB04-01
Date	1/13/2009
Depth Range (ft bgs)	1.0-3.0
TPH DRO and GRO (mg/kg)	
Total TPH	290 J

Site ID	60SB04
Sample ID	60-SB04-01D
Date	1/13/2009
Depth Range (ft bgs)	1.0-3.0
SVOCs (ug/kg)	
Benzo[a]pyrene	55 J
TPH DRO and GRO (mg/kg)	
Total TPH	440 J

Site ID	60SB04
Sample ID	60-SB04-03
Date	1/13/2009
Depth Range (ft bgs)	5.0-7.0
TPH DRO and GRO (mg/kg)	
Total TPH	840

Site ID	60SB03
Sample ID	60-SB03-01
Date	1/13/2009
Depth Range (ft bgs)	1.0-3.0
SVOCs (ug/kg)	
Benzo[a]pyrene	100
Benzo[b]fluoranthene	180
Dibenz(a,h)anthracene	29 J
Metals (mg/kg)	
Antimony	***120***
Arsenic	***33***
Copper	3,400 J
Lead	***1,600 J***
Zinc	2,600 J

Notes/Qualifiers:

ft bgs —feet below ground surface
ug/kg —micrograms per kilogram
mg/kg —milligrams per kilogram
J —Estimated value
BOLD —Exceeds Regional Residential Screening Criteria or PREQB Total TPH Screening Values
ITALICS —Exceeds Regional Screening Levels for Industrial Soil
UNDERLINE —Exceeds Basewide Background Screening Values for Metals

COWARDIAN WETLAND CLASSIFICATION

E2SS3 —ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.
M2US2 —MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

120 0 60 120
1 inch = 120 ft.

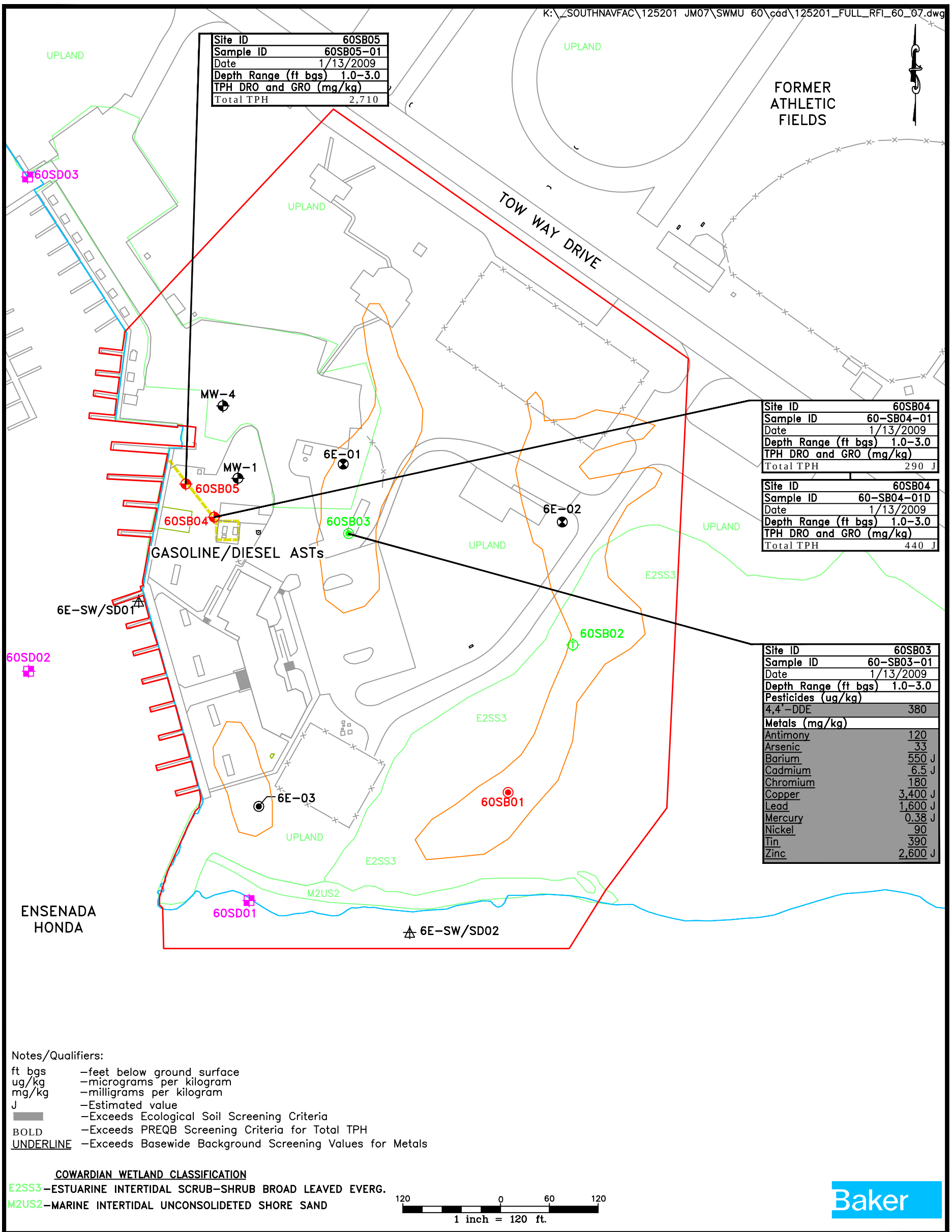
Baker

LEGEND

—SWMU BOUNDARY
—ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
—SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
—GASOLINE/DIESEL ASTs
—APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
—EXISTING MONITORING WELL LOCATION (1999 SITE CHARACTERIZATION)
—SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE II ECP 2004)
—SUBSURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (PHASE II ECP 2004)
—SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
—SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (TEMPORARY) (PHASE I RFI 2009)
—SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)

FIGURE 6
SUBSURFACE SOIL (1-7 ft bgs)
EXCEEDANCES OF HUMAN HEALTH AND BACKGROUND SCREENING CRITERIA-PHASE I RFI
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO



Notes/Qualifiers:

ft bgs	–feet below ground surface
ug/kg	–micrograms per kilogram
mg/kg	–milligrams per kilogram
J	–Estimated value
	–Exceeds Ecological Soil Screening Criteria
BOLD	–Exceeds PREQB Screening Criteria for Total TPH
UNDERLINE	–Exceeds Basewide Background Screening Values for Metals

COWARDIAN WETLAND CLASSIFICATION

E2SS3 –ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.

M2US2 –MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

120 0 60 120

1 inch = 120 ft.

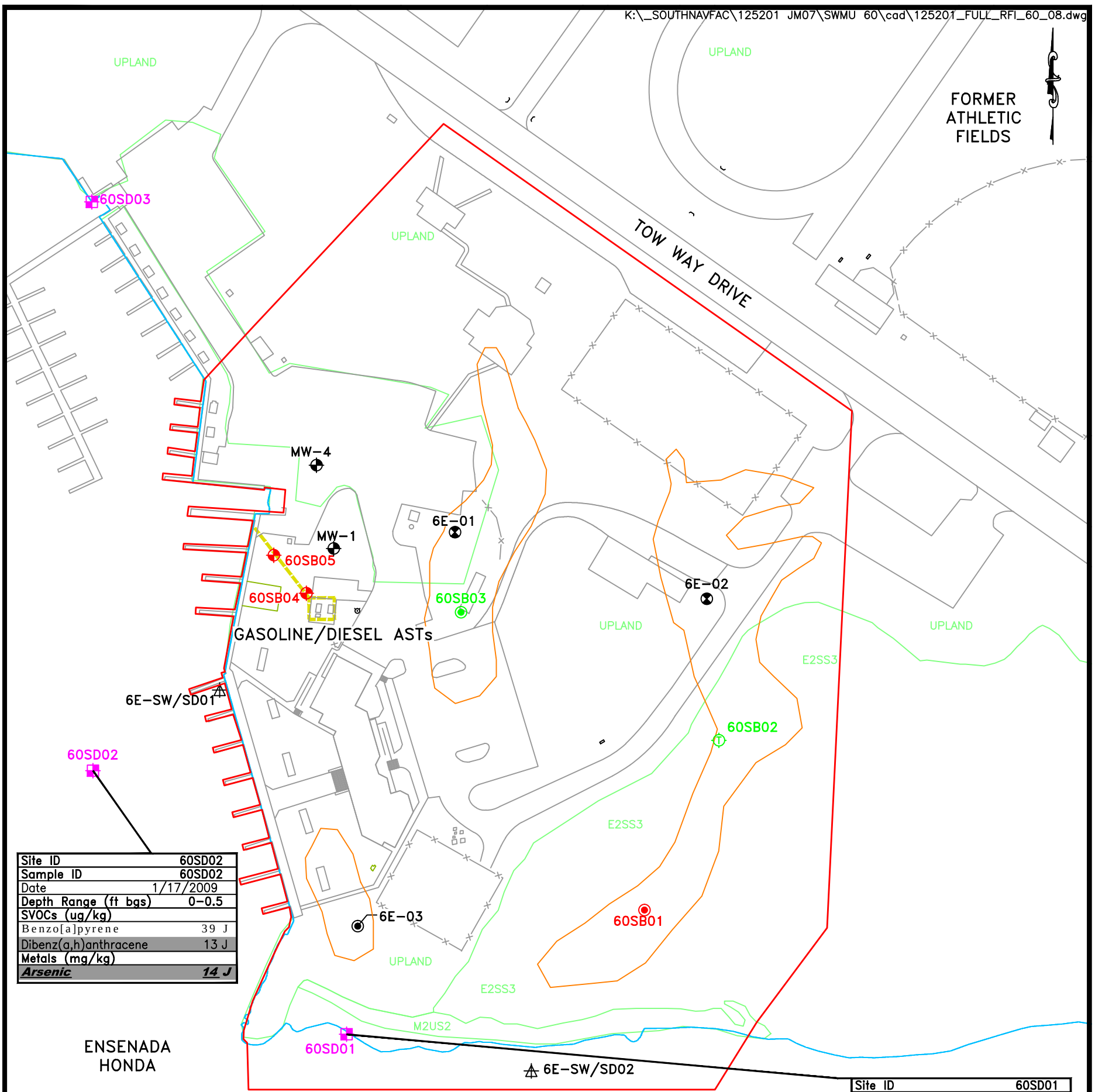
Baker

LEGEND

- –SWMU BOUNDARY
- –ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
- –SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
- –GASOLINE/DIESEL ASTs
- –APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
- –EXISTING MONITORING WELL LOCATION (1999 SITE CHARACTERIZATION)
- –SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE II ECP 2004)
- –SUBSURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (PHASE II ECP 2004)
- –SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
- –SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
- –SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
- –SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION (PHASE I RFI 2009)
- –SURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (TEMPORARY) (PHASE I RFI 2009)
- –SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)

FIGURE 7
SUBSURFACE SOIL (1-3 ft bgs)
EXCEEDANCES OF ECOLOGICAL AND
BACKGROUND SCREENING CRITERIA-PHASE I RFI
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO



Site ID	60SD02
Sample ID	60SD02
Date	1/17/2009
Depth Range (ft bgs)	0-0.5
SVOCs (ug/kg)	
Benzo[a]pyrene	39 J
Dibenz(a,h)anthracene	13 J
Metals (mg/kg)	
Arsenic	***14 J***

Site ID	60SD01
Sample ID	60SD01
Date	1/17/2009
Depth Range (ft bgs)	0.0-0.5
SVOCs (ug/kg)	
Acenaphthylene	30
Benzo[a]anthracene	210
Benzo[a]pyrene	***450***
Benzo[b]fluoranthene	700
Chrysene	470
Dibenz(a,h)anthracene	75
Dimethyl phthalate	14 J
Fluoranthene	370
Pyrene	950
Metals(mg/kg)	
Arsenic	***14***

Notes/Qualifiers:

ft bgs —feet below ground surface
ug/kg —micrograms per kilogram
mg/kg —milligrams per kilogram
J —Estimated value
—Exceeds Ecological Sediment Screening Criteria
BOLD —Exceeds Regional Screening Levels for Residential Soil
ITALICS —Exceeds Regional Screening Levels for Industrial Soil
UNDERLINE —Exceeds Basewide Background Screening Values for Metals

COWARDIAN WETLAND CLASSIFICATION

E2SS3 —ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.
M2US2 —MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

120 0 60 120
1 inch = 120 ft.

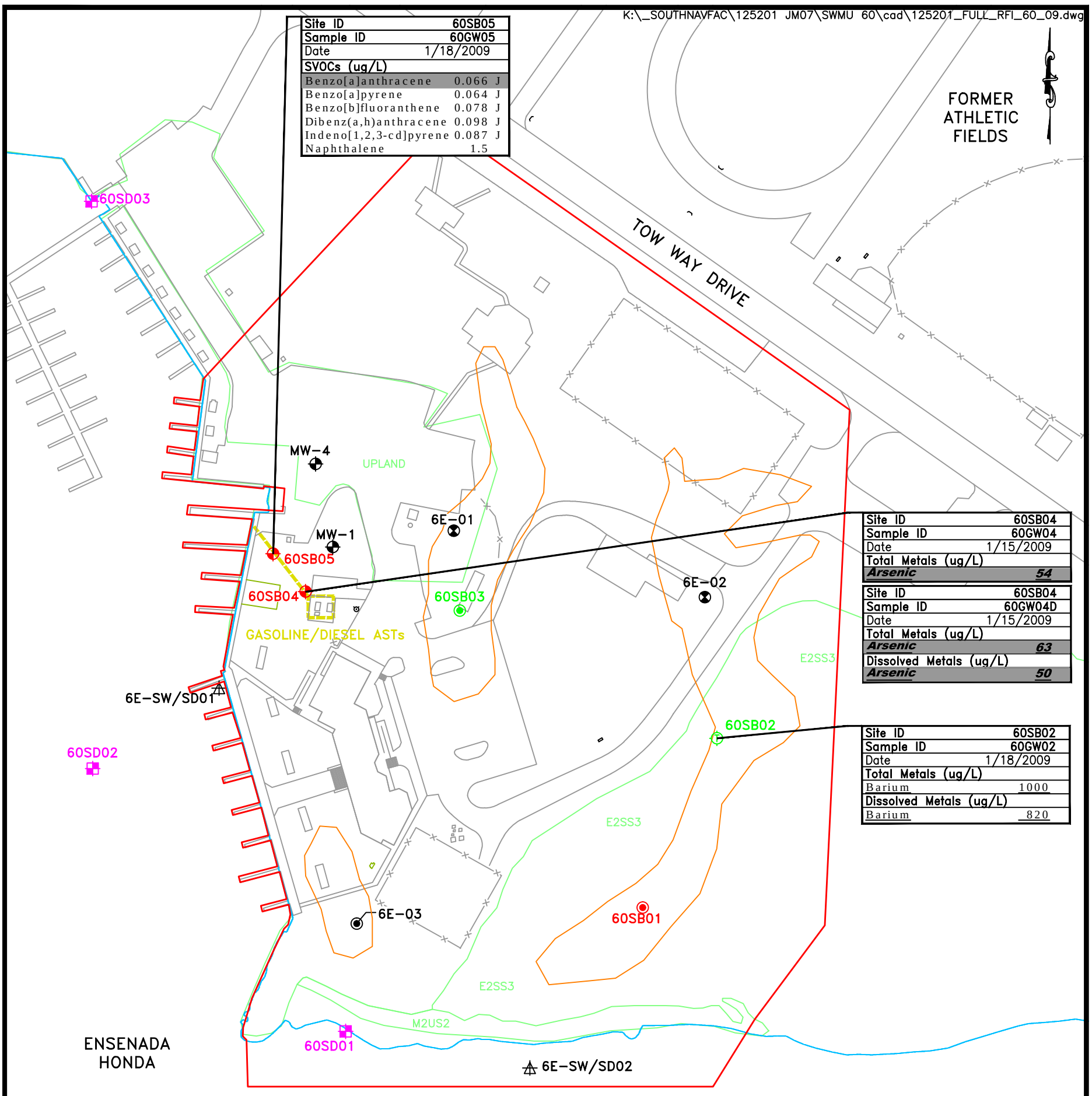
Baker

LEGEND

—SWMU BOUNDARY
—ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
—SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
—GASOLINE/DIESEL ASTs
—APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
—EXISTING MONITORING WELL LOCATION (1999 SITE CHARACTERIZATION)
—SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE II ECP 2004)
—SUBSURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (PHASE II ECP 2004)
—SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
—SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (TEMPORARY) (PHASE I RFI 2009)
—SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)

FIGURE 8
SEDIMENT EXCEEDANCES OF HUMAN HEALTH, ECOLOGICAL, AND BACKGROUND SCREENING CRITERIA-PHASE I RFI
SWMU 60-FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO



Site ID	60SB05
Sample ID	60GW05
Date	1/18/2009
SVOCs (ug/L)	
Benzo[a]anthracene	0.066 J
Benzo[a]pyrene	0.064 J
Benzo[b]fluoranthene	0.078 J
Dibenz(a,h)anthracene	0.098 J
Indeno[1,2,3-cd]pyrene	0.087 J
Naphthalene	1.5

Site ID	60SB04
Sample ID	60GW04
Date	1/15/2009
Total Metals (ug/L)	
Arsenic	***54***

Site ID	60SB04
Sample ID	60GW04D
Date	1/15/2009
Total Metals (ug/L)	
Arsenic	***63***
Dissolved Metals (ug/L)	
Arsenic	***50***

Site ID	60SB02
Sample ID	60GW02
Date	1/18/2009
Total Metals (ug/L)	
Barium	1000
Dissolved Metals (ug/L)	
Barium	820

Notes/Qualifiers:

ug/L —micrograms per kilogram
J —Estimated value
—Exceeds Ecological Groundwater Screening Criteria
BOLD —Exceeds Regional Tap Water Screening Level for Groundwater
ITALICS —Exceeds USEPA MCLs
UNDERLINE —Exceeds Basewide Background Screening Values for Metals

COWARDIAN WETLAND CLASSIFICATION

E2SS3 —ESTUARINE INTERTIDAL SCRUB-SHRUB BROAD LEAVED EVERG.
M2US2 —MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

120 0 60 120
1 inch = 120 ft.

Baker

LEGEND

—SWMU BOUNDARY
—ESTUARINE WETLAND BOUNDARY (GEO-MARINE, INC., 2000)
—SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
—GASOLINE/DIESEL ASTs
—APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
—EXISTING MONITORING WELL LOCATION (1999 SITE CHARACTERIZATION)
—SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE II ECP 2004)
—SUBSURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (PHASE II ECP 2004)
—SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
—SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION (PHASE I RFI 2009)
—SURFACE SOIL AND GROUNDWATER SAMPLE LOCATION (TEMPORARY) (PHASE I RFI 2009)
—SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)

FIGURE 9
GROUNDWATER EXCEEDANCES OF HUMAN HEALTH, ECOLOGICAL, AND BACKGROUND SCREENING CRITERIA—PHASE I RFI
SWMU 60—FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO

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UPLAND
UPLAND
UPLAND
UPLAND
UPLAND
UPLAND
E2SS3
E2SS3
E2SS3
M2US2
TOW WAY DRIVE
60SD03
MW-4
60SB14
60SB15
60SB06
60SB08
MW-1
60SB05
60SB07
60SB10
60SB12
60SB04
GASOLINE/DIESEL ASTs
60SB09
60SB11
60SB13
6E-01
6E-02
60SB03
60SB02
60SB01
60SD07
60SP01
6E-SW/SD01
60SD02
60SD08
60SD09
ENSENADA
HONDA
60SD04
60SD01
60SD06
6E-SW/SD02
60SD05

NOTES:
1. BORINGS 60SB16 THROUGH 60SB32 ARE NOT SHOWN. THE SPECIFIC NUMBER AND LOCATIONS OF THESE BORINGS WILL BE BASED ON RESULTS OF THE GEOPHYSICAL SURVEY.
2. APPROXIMATELY FOUR ADDITIONAL MONITORING WELLS WILL BE INSTALLED AND ARE NOT SHOWN. THE SPECIFIC NUMBER AND LOCATIONS OF THESE WELLS WILL BE BASED ON RESULTS OF THE GEOPHYSICAL SURVEY.

COWARDIAN WETLAND CLASSIFICATION
E2SS3 –ESTUARINE INTERTIDAL SCRUB–SHRUB BROAD LEAVED EVERG.
M2US2 –MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

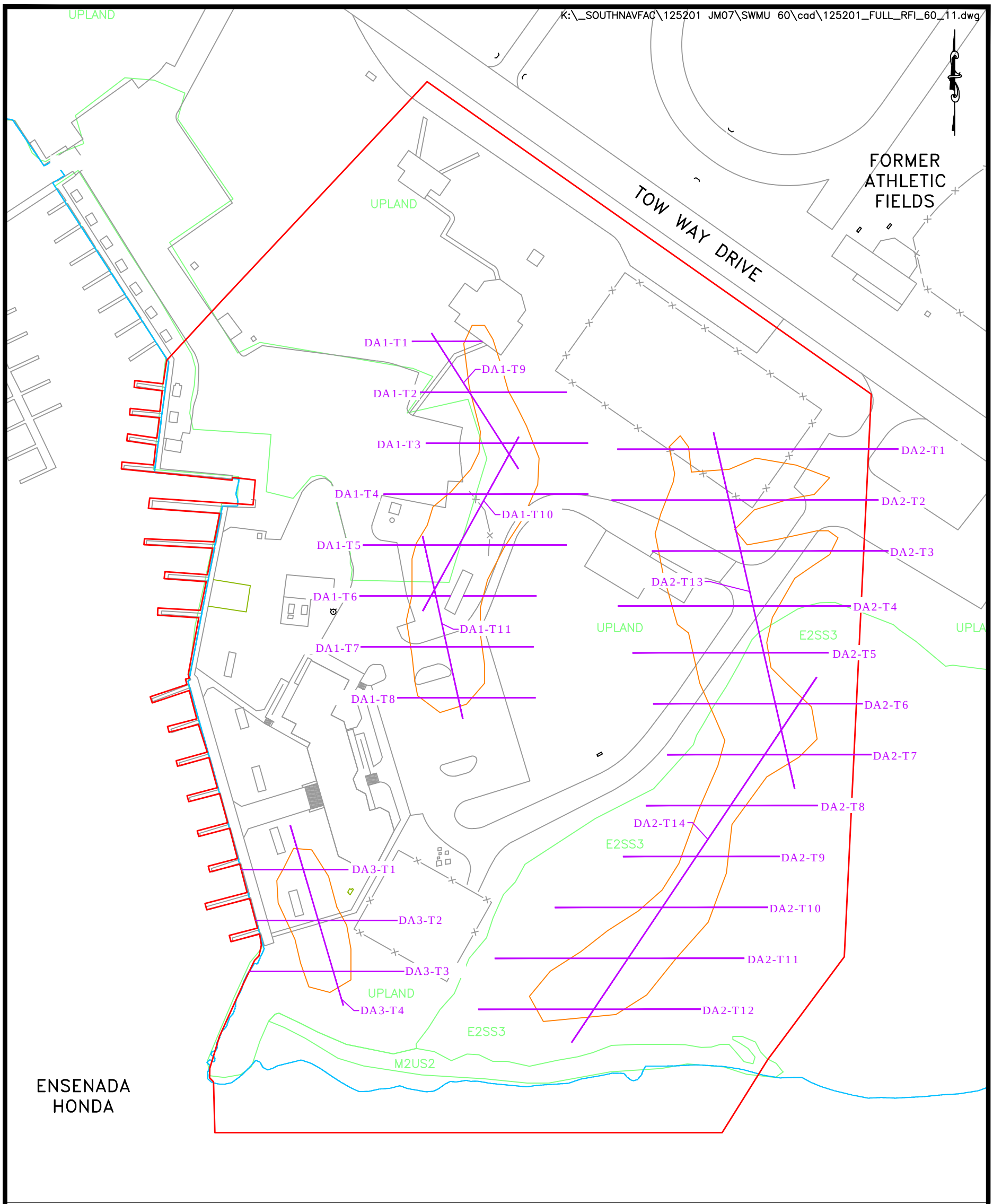
100 0 50 100
1 inch = 100 ft.

Baker

LEGEND
–SWMU BOUNDARY
–GASOLINE/DIESEL ASTs
–APPROXIMATE LOCATION OF FORMER UNDERGROUND PIPING SYSTEM
–ESTUARINE WETLAND BOUNDARY (GEO–MARINE, INC., 2000)
–SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)
–PRESUMED GROUNDWATER FLOW DIRECTION
–SURFACE WATER AND SEDIMENT SAMPLE LOCATION (PHASE II ECP 2004)
–SURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
–SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION (PHASE I RFI 2009)
–SEDIMENT SAMPLE LOCATION (PHASE I RFI 2009)
–EXISTING MONITORING WELL LOCATION (1999 SITE CHARACTERIZATION/PHASE I RFI)
–EXISTING MONITORING WELL LOCATION (PHASE II ECP 2004)
–EXISTING MONITORING WELL LOCATION (TEMPORARY) (PHASE I RFI 2009)
–PROPOSED SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION
–CONTINGENCY SURFACE AND SUBSURFACE SOIL SAMPLE LOCATION
–PROPOSED SURFACE, SUBSURFACE SOIL, AND GROUNDWATER SAMPLE LOCATION
–PROPOSED SEDIMENT SAMPLE LOCATION
–PROPOSED STAND PIPE LOCATION

FIGURE 10
PROPOSED SAMPLE AND MONITORING WELL LOCATION MAP
SWMU 60–FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO



COWARDIAN WETLAND CLASSIFICATION

E2SS3 –ESTUARINE INTERTIDAL SCRUB–SHRUB BROAD LEAVED EVERG.

M2US2 –MARINE INTERTIDAL UNCONSOLIDETED SHORE SAND

100 0 50 100

1 inch = 100 ft.

Baker

LEGEND

–SWMU BOUNDARY

–ESTUARINE WETLAND BOUNDARY (GEO–MARINE, INC., 2000)

–SUSPECTED DEBRIS DISPOSAL AREA (1958 POLYGON FEATURE; AERIAL PHOTO ANALYSIS, LANTDIV, 2004)

–PROPOSED GEOPHYSICAL SURVEY TRANSECT

FIGURE 11
PROPOSED GEOPHYSICAL SURVEY TRANSECT LOCATION MAP
SWMU 60–FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN

NAVAL ACTIVITY PUERTO RICO

APPENDIX A
SCREENING VALUES

SOIL

APPENDIX A

ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference	Soil to Groundwater SSLs [2]
Volatile Organics (µg/kg)					
1,1,1,2-Tetrachloroethane	1,900	9,300	100	CCME 2007	3.80
1,1,1-Trichloroethane	870,000 [3]	3,800,000 [3]	100	CCME 2007	1,400
1,1,2-Trichloroethane	1,100	5,300	100	CCME 2007	32.0
1,1-Dichloroethane	3,300	17,000	100	CCME 2007	13.6
1,1-Dichloroethene	24,000 [3]	110,000 [3]	100	CCME 2007	50.0
1,2,3-Trichloropropane	5.00	95.0	NE	NA	0.0056
1,2-Dibromo-3-chloropropane	5.40	69.0	NE	NA	1.72
1,2-Dibromoethane	34.0	170	300	CCME 2007	0.28
1,2-Dichloroethane	430	2,200	402	MHSPE 2000	28.0
1,2-Dichloropropane	940	4,700	700,000	Efroymson et al. 1997a	34.0
2-Butanone	2,800,000 [3]	20,000,000 [3]	NE	NA	20,000
2-Hexanone	21,000 [3]	140,000 [3]	NE	NA	158
3-Chloro-1-propene	680	3,400	NE	NA	4.00
4-Methyl-2-pentanone	530,000 [3]	5,300,000 [3]	NE	NA	4,600
Acetone	6,100,000 [3]	63,000,000 [3]	NE	NA	48,000
Acetonitrile	87,000 [3]	370,000 [3]	NE	NA	520
Acrolein	15.0 [3]	65.0 [3]	NE	NA	0.168
Acrylonitrile	240	1,200	1,000,000	Efroymson et al. 1997a	0.196
Benzene	1,100	5,400	101	MHSPE 2000	52.0
Bromodichloromethane	270	1,400	NE	NA	440
Bromoform	62,000	220,000	NE	NA	420
Bromomethane	730 [3]	3,200 [3]	NE	NA	36.0
Carbon Disulfide	82,000 [3]	370,000 [3]	NE	NA	4,200
Carbon Tetrachloride	610	3,000	1,000,000	Efroymson et al. 1997a	38.0
Chlorobenzene	29,000 [3]	140,000 [3]	40,000	Efroymson et al. 1997a	1,360
Chloroethane	1,500,000 [3]	6,100,000 [3]	NE	NA	118,000
Chloroform	290	1,500	1,002	MHSPE 2000	440

APPENDIX A

ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference	Soil to Groundwater SSLs [2]
Volatile Organics (µg/kg) (cont.)					
Chloromethane	12,000 [3]	50,000 [3]	NE	NA	980
Chloroprene	9.40	47.0	NE	NA	0.17
cis-1,3-Dichloropropene	1,700 [4]	8,300 [4]	100	CCME 2007	3.00
Dibromochloromethane	680	3,300	NE	NA	420
Dibromomethane	2,500 [3]	11,000 [3]	NE	NA	38.0
Dichlorodifluoromethane	9,400 [3]	40,000 [3]	NE	NA	6,000
Ethyl benzene	5,400	27,000	5,003	MHSPE 2000	15,600
Ethyl methacrylate	150,000 [3]	750,000 [3]	NE	NA	1,980
Iodomethane	NE	NE	NE	NA	NE
Isobutanol	1,800,000 [3]	18,000,000 [3]	NE	NA	19,000
Methacrylonitrile	320 [3]	1,800 [3]	NE	NA	3.40
Methyl methacrylate	480,000 [3]	2,100,000 [3]	NE	NA	6,000
Methylene Chloride	56,000	960,000	1,040	MHSPE 2000	26.0
Pentachloroethane	5,400	19,000	NE	NA	5.40
Propionitrile	NE	NE	NE	NA	NE
Styrene	630,000 [3]	3,600,000 [3]	10,030	MHSPE 2000	2,200
Tetrachloroethene	22,000	110,000	400	MHSPE 2000	46.0
Toluene	500,000 [3]	4,500,000 [3]	13,001	MHSPE 2000	13,800
trans-1,2-dichloroethene	15,000 [3]	69,000 [3]	100	CCME 2007	580
trans-1,3-Dichloropropene	1,700 [4]	8,300 [4]	100	CCME 2007	3.00
trans-1,4-Dichloro-2-butene	6.90	35.0	1,000,000	Efroymson et al. 1997a	0.0108
Trichloroethene	910	6,400	6,010	MHSPE 2000	36.0
Trichlorofluoromethane	79,000 [3]	340,000 [3]	NE	NA	13,800
Vinyl Acetate	97,000 [3]	410,000 [3]	NE	NA	1,740
Vinyl Chloride	60.0	1,700	11.0	MHSPE 2000	13.8
Xylene	59,000 [3]	250,000 [3]	1000	USEPA 2003	3,600

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ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference	Soil to Groundwater SSLs [2]
LLPAHs (µg/kg)					
2-Methylnaphthalene	23,000 [3]	220,000 [3]	NE	NA	2,800
Acenaphthene	340,000 [3]	3,300,000 [3]	NE	NA	82,000
Acenaphthylene	340,000 [3][5]	3,300,000 [3][5]	NE	NA	82,000 [5]
Anthracene	1,700,000 [3]	17,000,000 [3]	NE	NA	840,000
Benzo(a)anthracene	150	2,100	NE	NA	200
Benzo(a)pyrene	15.0	210	NE	NA	4,800
Benzo(b)fluoranthene	150	2,100	NE	NA	700
Benzo(g,h,i)perylene	170,000 [3][6]	1,700,000 [3][6]	NE	NA	190,000 [6]
Benzo(k)fluoranthene	1,500	21,000	NE	NA	7,000
Chrysene	15,000	210,000	NE	NA	22,000
Dibenzo(a,h)anthracene	15.0	210	NE	NA	220
Fluoranthene	230,000 [3]	2,200,000 [3]	NE	NA	1,400,000
Fluorene	230,000 [3]	2,200,000 [3]	NE	NA	80,000
Indeno(1,2,3-cd)pyrene	150	2,100	NE	NA	2,400
Naphthalene	3,600	18,000	NE	NA	9.40
Phenanthrene	170,000 [3][6]	1,700,000 [3][6]	NE	NA	190,000 [6]
Pyrene	170,000 [3]	1,700,000 [3]	NE	NA	190,000
LLPAH Totals (µg/kg)					
Low Molecular Weight PAHs	NE	NE	29,000	USEPA 2007a	NE
High Molecular Weight PAHs	NE	NE	18,000	USEPA 2007a	NE

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ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference	Soil to Groundwater SSLs [2]
Pesticides (µg/kg)					
4,4'-DDD	2,000	7,200	93.0	USEPA 2007b	1,320
4,4'-DDE	1,400	5,100	93.0	USEPA 2007b	920
4,4'-DDT	1,700	7,000	93.0	USEPA 2007b	1,340
Aldrin	29.0	100	401	MHSPE 2000	0.68
Alpha-BHC	77.0	270	201	MHSPE 2000	0.72
beta-BHC	270	960	3.98	USEPA 2003	2.60
Chlordane	1,600	6,500	224	USEPA 2003	2,800
Chlorobenzilate	4,400	16,000	NE	NA	17.6
delta-BHC	270 [7]	960 [7]	201	MHSPE 2000	2.60 [7]
Dieldrin	30.0	110	0.022	USEPA 2007c	1.22
Endosulfan I	37,000 [3][8]	370,000 [3][8]	100	Friday 1998	22,000 [8]
Endosulfan II	37,000 [3][8]	370,000 [3][8]	100	Friday 1998	22,000 [8]
Endosulfan sulfate	37,000 [3][8]	370,000 [3][8]	100	Friday 1998	22,000 [8]
Endrin	1,800 [3]	18,000 [3]	401	MHSPE 2000	1,620
Endrin Aldehyde	1,800 [3][9]	18,000 [3][9]	100	Friday 1998	1,620 [9]
gamma-BHC	520	2,100	5.00	USEPA 2003	24.0
Heptachlor	110	380	400	MHSPE 2000	660
Heptachlor epoxide	53.0	190	400	MHSPE 2000	82.0
Isodrin	NE	NE	3.32	USEPA 2003	NE
Kepone	49.0	170	100	Friday 1998	2.20
Methyoxychlor	31,000 [3]	310,000 [3]	100	Friday 1998	44,000
Toxaphene	440	1,600	100	Friday 1998	9,200

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ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference	Soil to Groundwater SSLs [2]
Metals (mg/kg)					
Antimony	3.10 [3]	41.0 [3]	10.0	USEPA 2005a	5.40
Arsenic	0.39	1.60	18.0	USEPA 2005b	5.80
Barium	1,500 [3]	19,000 [3]	330	USEPA 2005c	1,640
Beryllium	16.0 [3]	200 [3]	21.0	USEPA 2005d	64.0
Cadmium	7.00 [3]	80.0 [3]	0.77	USEPA 2005e	7.60
Chromium	12,000 [3][10]	150,000 [3][10]	26.0	USEPA 2008	3,600,000
Cobalt	2.30 [3]	30.0 [3]	13.0	USEPA 2005f	4.20
Copper	310 [3]	4,100 [3]	28.0	USEPA 2007d	920
Lead	400 [11]	800 [11]	11.0	USEPA 2005g	280
Mercury	1.00 [3]	4.30 [3]	0.10	Efroymson et al. 1997a	2.00
Nickel	150 [3]	2,000 [3]	38.0	USEPA 2007e	400
Selenium	39.0 [3]	510 [3]	0.52	USEPA 2007f	5.20
Silver	39.0 [3]	510 [3]	4.20	USEPA 2006	12.0
Thallium	0.078 [3]	1.00 [3]	1.00	Efroymson et al. 1997b	2.80
Tin	4,700 [3]	61,000 [3]	50.0	Efroymson et al. 1997b	46,000
Vanadium	39.0 [3]	520 [3]	7.80	USEPA 2005h	1,560
Zinc	2,300 [3]	31,000 [3]	46.0	USEPA 2007g	5,800
TPH DRO and GRO (mg/kg)					
Gasoline Range Organics	100 [12]	NE	NE	NA	NE
Diesel Range Organics	100 [12]	NE	NE	NA	NE

APPENDIX A

ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Notes:

LLPAHs - Low Level Polynuclear Aromatic Hydrocarbons
TPH - Total Petroleum Hydrocarbons
DRO - Diesel Range Organics
GRO - Gasoline Range Organics
SSLs - Soil Screning Levels

NA - Not Applicable
NE - Not Established
µg/kg - microgram per kilogram
mg/kg - milligram per kilogram

(1) May 2012 USEPA Regional Screening Levels

(2) Soil to groundwater SSLs are provided for informational purposes and are not considered project action limits. A dilution attenuation factor of 20 has been applied to the generic values given in USEPA Regional Screening Levels (May 2012).

(3) Noncarcinogenic Regional Screening Levels based on a target hazard quotient of 0.1 for conservative screening purposes.

(4) Value for 1,3-dichloroproene used as a surrogate.

(5) Value for Acenaphthene used as a surrogate.

(6) Value for Pyrene used as a surrogate.

(7) Value for Technical BHC used as a surrogate.

(8) Value for Endosulfan used as a surrogate.

(9) Value for Endrin used as a surrogate.

(10) Value for Chromium III used as a surrogate.

(11) USEPA Action Level for lead in soil.

(12) Puerto Rico Specific Value.

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ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

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USEPA. 2007g. Ecological Soil Screening Levels for Zinc (Interim Final). Office of Solid Waste and Emergency Response, Washington, D.C. OSWER Directive 9285.7-73.

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APPENDIX A

ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - SOIL
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVETIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

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GROUNDWATER

APPENDIX A

ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - GROUNDWATER
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACTILIY INVESTIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Tap Water [1]	MCL	PRWQS	Selected Ecological Screening Values	Ecological Screening Value Reference
Volatile Organics (µg/L)					
1,1,1,2-Tetrachloroethane	0.50	NE	NE	200	USEPA 2007
1,1,1-Trichloroethane	750.0 [2]	200	200	312	USEPA 2001
1,1,2-Trichloroethane	0.24	5.00	5.00	340	USEPA 2007
1,1-Dichloroethane	2.40	NE	NE	47.0	USEPA 2003
1,1-Dichloroethene	26.0 [2]	7.00	7.00	2,240	USEPA 2001
1,2,3-Trichloropropane	0.00065	NE	NE	274	USEPA 2007
1,2-Dibromo-3-chloropropane	0.00032	0.20	NE	100	USEPA 2007
1,2-Dibromoethane	0.0065	0.05	NE	48.0	USEPA 2007
1,2-Dichloroethane	0.15	5.00	3.80	1,130	USEPA 2001
1,2-Dichloropropane	0.38	5.00	5.00	2,400	USEPA 2001
2-Butanone	490 [2]	NE	NE	13,333	USEPA 2007
2-Hexanone	3.40 [2]	NE	NE	99.0	USEPA 2003
3-Chloro-1-propene	0.63	NE	NE	3.40	USEPA 2007
4-Methyl-2-pentanone	100 [2]	NE	NE	170	USEPA 2003
Acetone	1,200 [2]	NE	NE	1,000	USEPA 2007
Acetonitrile	13.0 [2]	NE	NE	12,000	USEPA 2003
Acrolein	0.0041 [2]	NE	190	0.55	USEPA 2001
Acrylonitrile	0.045	NE	0.51	58.1	USEPA 2007
Benzene	0.39	5.00	5.00	109	USEPA 2001
Bromodichloromethane	0.12	80.0	5.50	2,400	USEPA 2007
Bromoform	7.90	80.0	43.0	640	USEPA 2001
Bromomethane	0.70 [2]	NE	47.0	120	USEPA 2007
Carbon Disulfide	72.0 [2]	NE	NE	15.0	USEPA 2003
Carbon Tetrachloride	0.39	5.00	2.30	1,500	USEPA 2001
Chlorobenzene	7.20 [2]	100	100	105	USEPA 2001
Chloroethane	2,100 [2]	NE	NE	NE	NA

APPENDIX A

ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - GROUNDWATER
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACTILIY INVESTIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Tap Water [1]	MCL	PRWQS	Selected Ecological Screening Values	Ecological Screening Value Reference
Volatile Organics (µg/L) (cont.)					
Chloroform	0.19	80.0	57.0	815	USEPA 2001
Chloromethane	19.0 [2]	NE	NE	2,700	USEPA 2007
Chloroprene	0.016	NE	NE	NE	NA
cis-1,3-Dichloropropene	0.41 [3]	NE	NE	7.90	USEPA 2001
Dibromochloromethane	0.15	80.0	4.00	340	USEPA 2007
Dibromomethane	0.79 [2]	NE	NE	1,280	Buchman 2008
Dichlorodifluoromethane	19.0 [2]	NE	NE	1,280	Value for trichlorofluoromethane used as a surrogate
Ethyl benzene	1.30	700	530	4.30	USEPA 2001
Ethyl methacrylate	42.0 [2]	NE	NE	18,000	USEPA 2007
Iodomethane	NE	NE	NE	NE	NA
Isobutanol	460.0 [2]	NE	NE	10,000	USEPA 2007
Methacrylonitrile	0.075 [2]	NE	NE	NE	NA
Methyl methacrylate	140 [2]	NE	NE	2,800	USEPA 2003
Methylene Chloride	9.90	5.00	46.0	2,560	USEPA 2001
Pentachloroethane	0.56	NE	NE	56.2	Buchman 2008
Propionitrile	NE	NE	NE	15,200	USEPA 2007
Styrene	110 [2]	100	NE	170	USEPA 2007
Tetrachloroethene	9.70	5.00	5.00	45.0	USEPA 2001
Toluene	86.0 [2]	1,000	1,000	37.0	USEPA 2001
trans-1,2-dichloroethene	8.60 [2]	100	100	4,480	Buchman 2008
trans-1,3-Dichloropropene	0.41 [3]	NE	NE	7.90	USEPA 2001
trans-1,4-Dichloro-2-butene	0.0012	NE	NE	NA	NA
Trichloroethene	0.44	5.00	5.00	40.0	Buchman 2008
Trichlorofluoromethane	110 [2]	NE	NE	1,280	Buchman 2008
Vinyl Acetate	41.0 [2]	NE	NE	100	USEPA 2007
Vinyl Chloride	0.015	2.00	0.25	930	USEPA 2003
Xylene	19.0 [2]	NE	NE	27.0	USEPA 2003

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ECOLOGICAL AND HUMAN HEALTH, RISK-BASED SCREENING VALUES - GROUNDWATER
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACTILIY INVESTIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Tap Water [1]	MCL	PRWQS	Selected Ecological Screening Values	Ecological Screening Value Reference
LLPAHs (µg/L)					
2-Methylnaphthalene	2.70 [2]	NE	NE	6.00	USEPA 2007
Acenaphthene	40.0 [2]	NE	670	9.70	USEPA 2001
Acenaphthylene	40.0 [2][4]	NE	830 [1]	6.00	Buchman 2008
Anthracene	130 [2]	NE	8300	5.35	USEPA 2007
Benzo(a)anthracene	0.029	NE	0.038	0.025	USEPA 2003
Benzo(a)pyrene	0.0029	0.20	0.038	10.0	USEPA 2004
Benzo(b)fluoranthene	0.029	NE	0.038	6.00	Buchman 2008
Benzo(g,h,i)perylene	8.70 [2][5]	NE	830 [1]	6.00	Buchman 2008
Benzo(k)fluoranthene	0.29	NE	0.038	6.00	Buchman 2008
Chrysene	2.90	NE	0.038	10.0	USEPA 2004
Dibenzo(a,h)anthracene	0.0029	NE	0.038	6.00	Buchman 2008
Fluoranthene	63.0 [2]	NE	130	11.0	USEPA 1996
Fluorene	22.0 [2]	NE	1100	10.0	USEPA 2007
Indeno(1,2,3-cd)pyrene	0.029	NE	0.038	6.00	Buchman 2008
Naphthalene	0.14	NE	NE	23.5	USEPA 2001
Phenanthrene	8.70 [2][5]	NE	830 [1]	8.30	USEPA 1996
Pyrene	8.70 [2]	NE	830	0.248	USEPA 2007

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SWMU 60 - FORMER LANDFILL AT THE MARINA
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NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Tap Water [1]	MCL	PRWQS	Selected Ecological Screening Values	Ecological Screening Value Reference
Pesticides (µg/L)					
4,4'-DDD	0.28	NE	NE	0.001	PREQB 2010
4,4'-DDE	0.20	NE	NE	0.001	PREQB 2010
4,4'-DDT	0.20	NE	0.0022	0.001	PREQB 2010
Aldrin	0.00021	NE	0.00049	0.13	USEPA 2001
Alpha-BHC	0.0062	NE	0.026	2.30	USEPA 2007
beta-BHC	0.022	NE	0.091	32.0	USEPA 2007
Chlordane	0.027	2.00	NE	0.004	PREQB 2010
Chlorobenzilate	0.27	NE	NE	0.076	USEPA 2005
delta-BHC	0.022 [6]	NE	NE	0.125	USEPA 2007
Dieldrin	0.0015	NE	0.00052	0.0019	USEPA 2009
Endosulfan I	7.80 [2][7]	NE	62.0 [3]	0.0087	PREQB 2010
Endosulfan II	7.80 [2][7]	NE	62.0 [3]	0.0087	PREQB 2010
Endosulfan sulfate	7.80 [2][7]	NE	62.0	0.92	USEPA 2007
Endrin	0.17 [2]	2.00	0.059	0.0023	PREQB 2010
Endrin Aldehyde	0.17 [2][8]	2.00 [8]	0.29	NA	NA
gamma-BHC	0.036	0.20	0.20	0.16	PREQB 2010
Heptachlor	0.0018	0.40	0.00079	0.0036	USEPA 2009
Heptachlor epoxide	0.0033	0.20	0.20	0.0036	PREQB 2010
Isodrin	NE	NE	NE	0.12	USEPA 2007
Kepone	0.003	NE	NE	0.094	USEPA 2007
Methyoxychlor	2.70 [2]	40.0	40.0	0.03	PREQB 2010
Toxaphene	0.013	3.00	0.0028	0.0002	PREQB 2010

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Chemical	Regional Screening Levels Tap Water [1]	MCL	PRWQS	Selected Ecological Screening Values	Ecological Screening Value Reference
Metals (µg/L)					
Antimony	0.60 [2]	6.00	5.60	500	Buchman 2008
Arsenic	0.045	10.0	10.0	36.0	PREQB 2010
Barium	290 [2]	2,000	NE	16,667	USEPA 2007
Beryllium	1.60 [2]	4.00	NE	167	USEPA 2007
Cadmium	0.69 [2]	5.00	5.00	8.85	PREQB 2010
Chromium	1,600 [2][9]	100	100	50.4	PREQB 2010
Cobalt	0.47 [2]	NE	NE	45.0	USEPA 2007
Copper	62.0 [2]	1,300	1,300	3.73	PREQB 2010
Lead	15.0	15.0	15.0	8.52	PREQB 2010
Mercury	0.063 [2]	2.00	0.05	1.11	USEPA 2009
Nickel	30.0 [2]	NE	610	8.28	PREQB 2010
Selenium	7.80 [2]	50.0	50.0	71.1	PREQB 2010
Silver	7.10 [2]	NE	NE	2.24	PREQB 2010
Thallium	0.016 [2]	2.00	0.24	21.3	USEPA 2001
Tin	930 [2]	NE	NE	180	USEPA 2003
Vanadium	7.80 [2]	NE	NE	12.0	USEPA 2003
Zinc	470 [2]	NE	NE	85.6	PREQB 2010
TPH DRO and GRO (mg/L)					
Diesel Range Organics	50.0 [10]	NE	NE	NE	NA
Gasoline Range Organics	50.0 [10]	NE	NE	NE	NA

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Notes:

DRO - Diesel Range Organics
GRO - Gasoline Range Organics
LLPAHs - Low Level Polynuclear Aromatic Hydrocarbons
MCL - Maximum Contaminant Level
µg/L - microgram per liter
mg/L - milligram per liter
NA - Not Applicable
NE - Not Established
PRWQS - Puerto Rico Water Quality Standards
TPH - Total Petroleum Hydrocarbons

(1) May 2012 USEPA Regional Screening Levels
(2) Noncarcinogenic Regional Screening Levels based on a target hazard quotient of 0.1 for conservative screening purposes.
(3) Value for 1,3-dichloroproene used as a surrogate.
(4) Value for Acenaphthene used as a surrogate.
(5) Value for Pyrene used as a surrogate.
(6) Value for Technical BHC used as a surrogate.
(7) Value for Endosulfan used as a surrogate.
(8) Value for Endrin used as a surrogate.
(9) Value for Chromium III used as a surrogate.
(10) Puerto Rico Specific Value.

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SEDIMENT

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SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVESTIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference
LLPAHs (µg/kg)				
2-Methylnaphthalene	23,000 [2]	220,000 [2]	20.2	MacDonald 1994
Acenaphthene	340,000 [2]	3,300,000 [2]	6.71	MacDonald 1994
Acenaphthylene	340,000 [2][3]	3,300,000 [2][3]	5.87	MacDonald 1994
Anthracene	1,700,000 [2]	17,000,000 [2]	46.9	MacDonald 1994
Benzo(a)anthracene	150	2,100	74.8	MacDonald 1994
Benzo(a)pyrene	15.0	210	88.8	MacDonald 1994
Benzo(b)fluoranthene	150	2,100	1,800	Buchman 2008
Benzo(g,h,i)perylene	170,000 [2][4]	1,700,000 [2][4]	670	Buchman 2008
Benzo(k)fluoranthene	1,500	21,000	1,800	Buchman 2008
Chrysene	15,000	210,000	108	MacDonald 1994
Dibenzo(a,h)anthracene	15.0	210	6.22	MacDonald 1994
Fluoranthene	230,000 [2]	2,200,000 [2]	113	MacDonald 1994
Fluorene	230,000 [2]	2,200,000 [2]	21.2	MacDonald 1994
Indeno(1,2,3-cd)pyrene	150	2,100	600	Buchman 2008
Naphthalene	3,600	18,000	34.6	MacDonald 1994
Phenanthrene	170,000 [2][4]	1,700,000 [2][4]	86.7	MacDonald 1994
Pyrene	170,000 [2]	1,700,000 [2]	153	MacDonald 1994
Pesticides (µg/kg)				
4,4'-DDD	2,000	7,200	1.22	MacDonald 1994
4,4'-DDE	1,400	5,100	2.07	MacDonald 1994
4,4'-DDT	1,700	7,000	1.19	MacDonald 1994
Aldrin	29.0	100	9.50	Buchman 2008
Alpha-BHC	77.0	270	6.00	Persaud et al. 1993
beta-BHC	270	960	5.00	Persaud et al. 1993
Chlordane	1,600	6,500	2.26	--
Chlorobenzilate	4,400	16,000	34.7	USEPA 1993 and 1996
delta-BHC	270 [5]	960 [5]	6.96	USEPA 1993 and 1996
Dieldrin	30.0	110	0.72	MacDonald 1994
Endosulfan I	37,000 [2][6]	370,000 [2][6]	0.51	USEPA 1993 and 1996
Endosulfan II	37,000 [2][6]	370,000 [2][6]	0.51	USEPA 1993 and 1996

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Chemical	Regional Screening Levels Residential Soil [1]	Regional Screening Levels Industrial Soil [1]	Selected Ecological Screening Values	Ecological Screening Value Reference
Pesticides (µg/kg) (cont.)				
Endosulfan sulfate	37,000 [2][6]	370,000 [2][6]	36.5	USEPA 1993 and 1996
Endrin	1,800 [2]	18,000 [2]	2.22	MacDonald et al. 2000
Endrin Aldehyde	1,800 [2][7]	18,000 [2][7]	2.22	Value for Endrin used as surrogate
gamma-BHC	520	2,100	0.32	MacDonald 1994
Heptachlor	110	380	0.30	Buchman 2008
Heptachlor epoxide	53.0	190	2.47	MacDonald et al. 2000
Isodrin	NE	NE	2,944	USEPA 1993 and 1996
Kepone	49.0	170	153	USEPA 1993 and 1996
Methyoxychlor	31,000 [2]	310,000 [2]	29.6	USEPA 1993 and 1996
Toxaphene	440	1,600	0.10	MacDonald et al. 2000
Metals (mg/kg)				
Antimony	3.10 [2]	41.0 [2]	2.00	Long and Morgan 1991
Arsenic	0.39	1.60	7.24	MacDonald 1994
Barium	1,500 [2]	19,000 [2]	48.0	Buchman 2008
Beryllium	16.0 [2]	200 [2]	NE	NA
Cadmium	7.00 [2]	80.0 [2]	0.676	MacDonald 1994
Chromium	12,000 [2][8]	150,000 [2][8]	52.3	MacDonald 1994
Cobalt	2.30 [2]	30.0 [2]	10.0	Buchman 2008
Copper	310 [2]	4,100 [2]	18.7	MacDonald 1994
Lead	400 [9]	800 [9]	30.2	MacDonald 1994
Mercury	1.00 [2]	4.30 [2]	0.13	MacDonald 1994
Nickel	150 [2]	2,000 [2]	15.9	MacDonald 1994
Selenium	39.0 [2]	510 [2]	1.00	Buchman 2008
Silver	39.0 [2]	510 [2]	0.733	MacDonald 1994
Thallium	0.078 [2]	1.00 [2]	NE	NA
Tin	4,700 [2]	61,000 [2]	3.40	Buchman 2008
Vanadium	39.0 [2]	520 [2]	57.0	Buchman 2008
Zinc	2,300 [2]	31,000 [2]	124	MacDonald 1994

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SWMU 60 - FORMER LANDFILL AT THE MARINA
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Notes:

LLPAHs - Low Level Polynuclear Aromatic Hydrocarbons
NA - Not Applicable
NE - Not Established
µg/kg - microgram per kilogram
mg/kg - milligram per kilogram

(1) May 2012 USEPA Regional Screening Levels.
(2) Noncarcinogenic Regional Screening Levels based on a target hazard quotient of 0.1 for conservative screening purposes.
(3) Value for Acenaphthene used as a surrogate.
(4) Value for Pyrene used as a surrogate.
(5) Value for Technical BHC used as a surrogate.
(6) Value for Endosulfan used as a surrogate.
(7) Value for Endrin used as a surrogate.
(8) Value for Chromium III used as a surrogate.
(9) USEPA Action Level for lead in soil.

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RATIONALE FOR SELECTING ECOLOGICAL SCREENING VALUES

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RATIONALE FOR SELECTING ECOLOGICAL SCREENING VALUES
SWMU 60 – FORMER LANDFILL AT THE MARINA
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Soil Screening Values

USEPA Ecological Soil Screening Levels (Eco-SSLs) were preferentially used as soil screening values (documentation available at http://www.epa.gov/ecotox/ecossl/). Eco-SSLs have been developed for eight receptor groups: plants, soil invertebrates, avian herbivores, avian ground insectivores, avian carnivores, mammalian herbivores, mammalian ground insectivores, and mammalian carnivores. For a given chemical, the lowest Eco-SSL value for plants, soil invertebrates, avian herbivores, avian ground insectivores, avian carnivores, and mammalian herbivores was selected as the soil screening value. Eco-SSLs for mammalian ground insectivores were not considered for soil screening value development because there are no mammalian ground insectivores in Puerto Rico (mammalian insectivores are limited to aerial insectivores [i.e., bats]). As discussed in Guidelines for Developing Ecological Soil Screening Levels (USEPA, 2005), aerial and arboreal insectivorous birds and mammals were excluded from consideration during Eco-SSL development because they are considered inappropriate (i.e., they do not have a clear or indirect exposure pathway link to soil [indirect exposure pathways involve ingestion of prey that have direct contact with soil]). Eco-SSLs for mammalian carnivores were also not considered for soil screening value development because there are no carnivorous mammals on Puerto Rico. With the exception of bats, the terrestrial mammals represented by potentially complete exposure pathways are limited to nonindigenous, nuisance species (e.g., Norway rat, black rat, and mongoose) that have been implicated in the decline of native reptilian and bird populations (Mac et al., 1998 and United States Fish and Wildlife Service [USFWS], 1996). Eco-SSLs for mammalian herbivores are considered appropriate for soil screening value development based on the presence of fruit-eating and nectivorous bats in Puerto Rico (Gannon et al., 2005).

For those chemicals lacking plant, soil invertebrate, avian herbivore, avian ground insectivore, avian carnivore, and mammalian herbivore Eco-SSLs, the literature-based toxicological benchmarks listed below were used as soil screening values.

- USEPA Region 5 (2003) ecological screening levels (ESLs) for soil based on exposures to plants or invertebrates
- Toxicological thresholds for earthworms and microorganisms (Efroymson et al., 1997a)
- Toxicological thresholds for plants (Efroymson et al., 1997b)

Identical to the Eco-SSLs, if more than one screening value was available for a given chemical from USEPA (2003) and Efroymson et al. (1997a and 1997b), the lowest value was selected as the soil screening value.

For those chemicals lacking an Eco-SSL, toxicological data eligible for Eco-SSL derivation, USEPA Region 5 ESLs based on exposures to plants or terrestrial invertebrates, and a toxicological threshold from Efroymson et al. (1997a and 1997b), the following literature-based values, listed in their order of decreasing preference, were used as soil screening values:

- Toxicity reference values for plants and invertebrates listed in USEPA (1999)
- Soil standards developed by the Ministry of Housing, Spatial Planning and Environment (MHSPE, 2000)
- Canadian soil quality guidelines (agricultural land use) developed by the Canadian Council of Ministers of the Environment (CCME, 2001 and 2007)
- Ecological soil screening values compiled by Friday (1998)

Soil screening values based on MHSPE soil standards represent an average of the target and intervention soil standards. Values are based on a default organic carbon content of 2.0 percent, which represents the minimum value within the adjustment range (2.0 to 30.0 percent). Soil quality guidelines developed by CCME (2001 and 2007), as well as ecological soil screening values compiled by Friday (1998) were given the lowest preference since many are based on background concentrations or detection limits, not effect-based concentrations.

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Groundwater Screening Values

Puerto Rico Water Quality Standards (PRWQS) for Class SB coastal and estuarine waters listed in the Puerto Rico Water Quality Standards Regulation (PRWQSR) dated March 31, 2010 (PREQB, 2010) were preferentially used as groundwater screening values. PRWQS for Class SB coastal and estuarine waters were selected based on the classifications contained within Rule 1302.1 of the PRWQSR. For those chemicals lacking PRWQS for Class SB coastal and estuarine waters, groundwater screening values were identified from the following information listed in their order of decreasing preference:

- Chronic saltwater NAWQC (USEPA, 2009)
- Final Chronic Values (FCVs) for saltwater contained in ECO Update Volume 3, Number 2 (USEPA, 1996)
- USEPA Region 4 chronic screening values for saltwater contained in Ecological Risk Assessment Bulletins – Supplement to Risk Assessment Guidance for Superfund (RAGS) (USEPA 2001)
- Minimum chronic toxicity test endpoints (No Observed Effect Concentration [NOEC], No Observed Effect Level [NOEL], and MATC values based on reproduction, growth, or survival) for marine species reported in the ECOTOXicology (ECOTOX) Release 4.0 Database System (USEPA, 2007)
- Chronic Lowest Observable Effect Levels (LOELs) for saltwater contained in National Oceanic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQUIRTs) (Buchman, 2008) with a safety factor of 5 (Wentsel et al., 1996)

The order of preference was selected based on their level of protection. For example, NAWQC and FCVs would be expected to offer a greater degree of protection than a single species NOEC, MATC, or LOEL since their derivation considers a larger toxicological database. In the absence of the above-mentioned NAWQC, FCVs, USEPA Region 4 chronic screening values, chronic test endpoints (NOEC, NOEL, and MATC values), and chronic LOELs, screening values were derived from the literature-based acute saltwater values listed below:

- Acute LOELs for saltwater contained in NOAA SQUIRTs (Buchman, 2008)
- Acute toxicity test endpoints (NOEC, NOEL, LOEL, Lowest Observed Effect Concentration [LOEC], median lethal concentration [LC_{50}], and median effective concentration [EC_{50}] values) for marine species contained in the ECOTOX Release 4.0 Database System (USEPA, 2007)
- LC_{50} values for marine species contained in Superfund Chemical Matrix (USEPA, 2004)

Chronic-based screening values were extrapolated from acute NOEC, NOEL, LOEC, LOEL, LC_{50}, and EC_{50} values as follows:

- A safety factor of 30 was used to convert an acute NOEC or NOEL to a chronic-based screening value (Wentsel et al., 1996)

- A safety factor of 50 was used to convert an acute LOEC or LOEL to a chronic-based screening value (Wentsel et al., 1996)

- A safety factor of 100 was used to convert an EC_{50} or LC_{50} to a chronic-based screening value (Wentsel et al., 1996)

When acute toxicity data were used to extrapolate a chronic screening value, NOECs/NOELs were given preference over LOECs/LOELs, LOECs/LOELs were given preference over LC_{50} and EC_{50} values, and EC_{50} values were given preference over LC_{50} values. When more than one value was available from the literature for a given test endpoint (e.g., NOEC), the minimum value was conservatively used to extrapolate a chronic screening value.

The screening value selected for mercury is an USEPA saltwater NAWQC (i.e., criteria continuous concentration [CCC]). The saltwater CCC value for this metal is identified in National Recommended Water Quality Criteria (USEPA, 2009) as a dissolved concentration. A total recoverable CCC value for mercury was derived for use as a groundwater screening value in the Step 2 screening level risk calculation by dividing the dissolved CCC value (0.94 µg/L) by 0.85 (saltwater conversion factor for mercury listed in Appendix A of National Recommended Water Quality Criteria [USEPA, 2009]).

For those chemicals lacking saltwater toxicological thresholds and literature values, groundwater screening values were identified or developed from the literature-based freshwater values listed below in their order of decreasing preference.

- PRWQS for Class SD surface waters listed in the PRWQSR (PREQB, 2010).

- Chronic freshwater NAWQC (USEPA, 2009)

- FCVs for freshwater contained in ECO Update Volume 3, Number 2 (USEPA, 1996)

- USEPA Region 4 chronic screening values for freshwater contained in Ecological Risk Assessment Bulletins – Supplement to RAGs (USEPA 2001) and USEPA Region 5 ESLs for surface water (USEPA, 2003)

- Minimum chronic toxicity test endpoints (NOEC, NOEL, and MATC values) for freshwater species reported in the ECOTOX Database System (USEPA, 2007)

- Great Lakes basin Tier II Secondary Chronic Values (SCVs) listed in the Great Lakes Initiative Toxicity Data Clearinghouse (http://www.epa.gov/gliclearinghouse/) (USEPA, 2011)

- Chronic LOELs for freshwater contained in NOAA SQUIRTs (Buchman, 2008) with a safety factor of 10 (Wentsel et al., 1996)

Identical to saltwater-based values, the order of preference was selected based on their level of protection. It is noted that USEPA Region 4 and Region 5 screening values were given equal preference. When a

value was available from both sources, the minimum value was selected as the surface water screening value. In the absence of the above-mentioned freshwater PRWQS, NAWQC, FCVs, USEPA Region 4 and Region 5 screening values, chronic test endpoints, Great lakes basin Tier II SCVs, and chronic LOELs, screening values were derived from the acute literature values listed below:

- Acute LOELs for freshwater contained in NOAA SQUIRTs (Buchman, 2008)
- Acute toxicity test endpoints (NOEC, NOEL, LOEL, LOEC, LC_{50}, EC_{50} values) for freshwater species contained in the ECOTOX Database System (USEPA, 2007)
- LC_{50} values for freshwater species contained in Superfund Chemical Matrix (USEPA, 2004)

Chronic-based screening values were extrapolated from acute NOEC, NOEL, LOEC, LOEL, LC_{50}, and EC_{50} values using the safety factors from Wentsel et al. (1996) identified above.

When acute toxicity data were used to extrapolate a chronic screening value, NOECs/NOELs were given preference over LOECs/LOELs, LOECs/LOELs were given preference over LC_{50} and EC_{50} values, and EC_{50} values were given preference over LC_{50} values. When more than one value was available from the literature for a given test endpoint (e.g., NOEC), the minimum value was conservatively used to extrapolate a chronic screening value. In some cases, acute and/or chronic saltwater and freshwater LOELs for chemical classes (e.g., PAHs) were available from the literature (Buchman, 2008). A LOEL based on a chemical class was selected as the screening value only if that chemical lacks saltwater and freshwater literature-based benchmarks and/or toxicity test endpoints.

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Sediment Screening Values

The marine and estuarine bulk sediment toxicological benchmarks listed below were preferentially used as sediment screening values:

- Effects-Range low (ER-L) marine and estuarine sediment quality guidelines (SQGs) (Long and Morgan, 1991 and Long et al., 1995).

- Threshold Effects Level (TEL) marine sediment quality assessment guidelines (SQAGs) (MacDonald, 1994).

- Apparent Effects Threshold (AET) marine SQGs (Buchman, 2008).

A description of ER-L, TEL, and AET values and the methods used in their derivation are provided in the paragraphs that follow.

ER-L marine and estuarine SQGs. Long and Morgan (1991) developed effects-based SQGs using literature-based data from Equilibrium Partitioning (EqP) modeling, spiked-sediment toxicity tests, and matched sediment chemistry and biological effects measures. For a given chemical, the data were arranged in ascending order of concentration with each data entry assigned an "effects" or "no effects" descriptor, and the 10th percentile and 50th percentile concentrations of the "effects" data were calculated. The 10th and 50th percentiles of the "effects" data represent the ER-L and Effects Range-Median (ER-M), respectively. The ER-L and the ER-M delineate three concentration ranges for a given chemical. The concentration range below the ER-L value represents a minimal effects range (i.e., the concentration range in which effects would be rarely observed). Concentrations equal to or greater than the ER-L, but less than the ER-M represent a possible effects range within which effects would occasionally occur, while concentrations greater than the ER-M represent a probable-effects range within which effects would frequently occur. The ER-L and ER-M values were recalculated by Long et al. (1995) after omitting a small amount of freshwater data included in the original calculations (Long and Morgan, 1991) and incorporating more recent marine and estuarine data from the literature. With the exception of antimony, ER-Ls based on marine only SQGs from Long et al. (1995) were considered for use as sediment screening values. In the case of antimony, an ER-L value is not available from Long et al. (1995). Therefore, the ER-L value reported by Long and Morgan (1991) was considered as a potential sediment screening value.

TEL marine SQAGs for Florida coastal waters. The updated and revised data set used by Long et al. (1995) also was used by MacDonald (1994) to calculate SQAGs for Florida coastal waters (TELs and Probable Effect Levels [PELs]). Unlike the methodology used by Long et al. (1995) to derive ER-L and ER-M values, the derivation of TELs and PELs took into consideration the "no effects" data set. Specifically, TELs were derived by calculating the geometric mean of the 15th percentile in the "effects" data set and the 50th percentile in the "no effects" data set, while PELs were derived by calculating the geometric mean of the 50th percentile in the "effects" data set and the 85th percentile in the "no effects" data set.

Identical to ER-Ls and ER-Ms, TELs and PELs delineate three concentration ranges for a given chemical. The TEL represents the upper limit of the range of sediment concentrations dominated by "no effects" data. Within this range, concentrations are not considered to represent significant hazards to sediment-associated biota. The PEL represents the lower limit of the range of sediment concentrations that are usually or always associated with adverse biological effects. The range of concentrations that could be associated with biological effects is delineated by the TEL and PEL. Within this range of concentrations, adverse biological effects are possible. Only TELs were considered for use as sediment screening values.

AET marine SQGs. The AET method, developed by Tetra Tech, Inc (1986), associates chemical concentrations in sediments with adverse biological effects (lethal and sub-lethal toxicity as measured using sediment toxicity tests or changes in benthic macroinvertebrate abundance and community structure as measured by *in situ* biological surveys). For a given chemical and measurement of biological effect (biological indicator), the AET value represents the sediment concentration above which statistically significant biological effects are always observed. The AET values shown in Table 5-6 represent minimum AET values from a suite of seven biological indicators (amphipod mortality, oyster larval abnormality, Microtox luminescence, benthic macroinvertebrate abundance, bivalve larvae mortality/abnormality, Echinoderm larvae mortality/abnormality, and juvenile polychaete growth). It is noted that the AET values developed by Buchman (2008) are interim values subject to change.

Minimum, chemical-specific AET values are used by the Washington State Department of Ecology (1995) as sediment management standards for Puget Sound. Minimum AET values also are used by the United States Army Corps of Engineers (USACE) (USEPA/USACE, 1998) as "reason to believe" guidance for screening levels for the Dredged Material Management Program (DMMP). The DMMP screening levels are implemented for use in Puget Sound and Grays Harbor/Willapa Bay in the State of Washington. Current Washington State Department of Ecology sediment management standards and USACE DMMP screening levels do not reflect the interim AET values reported by Buchman (2008).

For a given chemical, when more than one toxicological threshold was available from the sources listed above (i.e., Long et al., 1995, MacDonald, 1994, and Buchman, 2008), the minimum value was conservatively selected as the sediment screening value. For those organic chemicals lacking literature-based marine and estuarine toxicological benchmarks, EqP-based screening values were either developed using USEPA methodology (USEPA, 1993 and 1996 or identified from the literature (Di Toro and McGrath, 2000). For a given chemical, when an EqP-based value was derived in accordance with USEPA (1993 and 1996) methodology and a value also was available from Di Toro and McGrath (2000), the minimum value was selected as the sediment screening value. As discussed in Appendix E, EqP-based screening values developed in accordance with USEPA (1993 and 1996) methodology are based, in part, on the fraction of organic carbon (f_{oc}) measured in sediment. The EqP-based screening values listed in Table 5-6 are based on a default f_{oc} of 0.01. As sediment samples collected from the estuarine wetlands contiguous to SWMUs 27 will be analyzed for TOC (see Table 3-1), the EqP-based screening values listed in Table 5-6 will be adjusted to reflect site-specific f_{oc} values. For the SERA, the minimum f_{oc} measured in SWMU 27 sediment will be used in the adjustment. The minimum f_{oc} value also was used to adjust the Di Toro and McGrath (2000) EqP-based toxicological benchmarks selected as sediment screening values for chloroethane, trans-1,2-dichloroethene, 4-bromophenyl phenyl ether, and 4-chlorophenyl phenyl ether (published values for these four organics are based on a default f_{oc} of 0.01).

It is noted that consideration was given to the following literature-based freshwater toxicological thresholds for chemicals lacking marine and estuarine bulk sediment values: (1) consensus-based SQGs for freshwater (MacDonald et al., 2000), (2) SQAGs for Florida inland waters (MacDonald et al., 2003), (3) Ontario Ministry of the Environment Lowest Effect Level (LEL) Provincial sediment quality guidelines (PSQGs) (Persaud et al., 1993), and (4) Canadian interim freshwater sediment quality

guidelines (ISQGs) (CCME, 2002). However, no values for chemicals lacking marine and estuarine bulk sediment toxicological thresholds were available from these sources.

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APPENDIX A

ECOLOGICAL GROUNDWATER SCREENING VALUES
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVESTIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Groundwater Screening Value [1]	Reference	Comment [2]
Volatile Organics (µg/L):			
1,1,1,2-Tetrachloroethane	200 [3]	USEPA 2007	Minimum acute value (96-hour LC_{50} for *Lepomis macrochirus* [bluegill]) with a safety factor of 100
1,1,1-Trichloroethane	312	USEPA 2001	USEPA Region 4 chronic screening value
1,1,2,2-Tetrachloroethane	90.2	USEPA 2001	USEPA Region 4 chronic screening value
1,1,2-Trichloroethane	340	USEPA 2007	Minimum acute value (48-hr LC_{50} for *Pleuronectes platessa* [sand dab]) with a safety factor of 100
1,1-Dichloroethane	47.0 [3]	USEPA 2003	USEPA Region 5 ecological screening level
1,1-Dichloroethene	2,240	USEPA 2001	USEPA Region 4 chronic screening value
1,2,3-Trichloropropane	274 [3]	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Pimephales promelas* [fathead minnow]) with a safety factor of 100
1,2-Dibromo-3-chloropropane	100	USEPA 2007	Minimum acute value (48-hr EC_{50} for *Mercenaria mercenaria* [hard clam]) with a safety factor of 100
1,2-Dibromoethane	48.0	USEPA 2007	Minimum acute value (48-hr LC_{50} for *Cyprinodon variegatus* [sheepshead minnow]) with a safety factor of 100
1,2-Dichloroethane	1,130	USEPA 2001	USEPA Region 4 chronic screening value
1,2-Dichloropropane	2,400	USEPA 2001	USEPA Region 4 chronic screening value
2-Butanone (MEK)	13,333	USEPA 2007	Minimum acute value (96-hour NOEC for *Cyprinodon variegatus* [sheepshead minnow]) with a safety factor of 30
2-Hexanone	99.0 [3]	USEPA 2003	USEPA Region 5 ecological screening level
3-Chloro-1-propene	3.40 [3]	USEPA 2007	Minimum acute value (48-hr LC_{50} for *Xenopus laevis* [clawed toad]) with a safety factor of 100
4-Methyl-2-pentanone (MIBK)	170 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Acetone	1,000	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Lumbriculus variegatus* [Oligochaete]) with a safety factor of 100
Acetonitrile	12,000 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Acrolein	0.55	USEPA 2001	USEPA Region 4 chronic screening value
Acrylonitrile	58.1	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Americamysis bahia* [opossum shrimp]) with a safety factor of 100
Benzene	109	USEPA 2001	USEPA Region 4 chronic screening value
Bromodichloromethane	2,400 [3]	USEPA 2007	Minimum acute value (24-hr LC_{50} for *Tetrahymena pyriformis* [ciliate]) with a safety factor of 100
Bromoform	640	USEPA 2001	USEPA Region 4 chronic screening value
Bromomethane	120	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Menidia beryllina* [inland silverside]) with a safety factor of 100
Carbon disulfide	15.0 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Carbon tetrachloride	1,500	USEPA 2001	USEPA Region 4 chronic screening value
Chlorobenzene	105	USEPA 2001	USEPA Region 4 chronic screening value
Chloroethane	NA	---	---
Chloroform	815	USEPA 2001	USEPA Region 4 chronic screening value
Chloromethane	2,700	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Menidia beryllina* [inland silverside]) with a safety factor of 100
Chloroprene	NA	---	---
cis-1,3-Dichloropropene	7.90	USEPA 2001	USEPA Region 4 chronic screening value (cis and trans)
Dibromochloromethane	340 [3]	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Cyprinus carpio* [common carp] with a safety factor of 100
Dibromomethane	1,280	Buchman 2008	Chronic LOEL for chemical class with a safety factor of 5
Dichlorodifluoromethane	1,280	---	Value for trichlorofluoromethane used as a surrogate
Ethyl benzene	4.30	USEPA 2001	USEPA Region 4 chronic screening value
Ethyl methacrylate	18,000 [3]	USEPA 2007	Minimum chronic value (21-day NOEC for *Daphnia magna* [cladoceron] based on reproduction [progeny counts])
Iodomethane	NA	---	---
Isobutanol	10,000	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Alburnus alburnus* [bleak]) with a safety factor of 100
Methacrylonitrile	NA	---	---

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Chemical	Groundwater Screening Value [1]	Reference	Comment [2]
Volatile Organics (µg/L) (cont.):			
Methyl methacrylate	2,800 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Methylene chloride	2,560	USEPA 2001	USEPA Region 4 chronic screening value
Pentachloroethane	56.2	Buchman 2008	Chronic LOEL with a safety factor of 5
Propionitrile	15,200 [3]	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Pimephales promelas* [fathead minnow]) with a safety factor of 100
Styrene	170	USEPA 2007	Minimum acute value (96-hr NOEC for *Cyprinodon variegatus* [sheepshead minnow]) with a safety factor of 30
Tetrachloroethene	45.0	USEPA 2001	USEPA Region 4 chronic screening value
Toluene	37.0	USEPA 2001	USEPA Region 4 chronic screening value
trans-1,2-dichloroethene	4,480	Buchman 2008	Acute LOEL (summation of all isomers) with a safety factor of 50
trans-1,3-Dichloropropene	7.90	USEPA 2001	USEPA Region 4 chronic screening value (cis and trans)
trans-1,4-Dichloro-2-butene	NA	---	---
Trichloroethene	40.0	Buchman 2008	Acute LOEL with a safety factor of 50
Trichlorofluoromethane	1,280	Buchman 2008	Chronic LOEL for chemical class with a safety factor of 5
Vinyl acetate	100	USEPA 2007	Minimum acute value (48-hr LC_{50} for *Crangon crangon* [sand shrimp]) with a safety factor of 100
Vinyl chloride	930 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Xylenes (total)	27.0 [3]	USEPA 2003	USEPA Region 5 ecological screening level
LLPAHs (µg/L):			
2-Methylnaphthalene	6.00	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Penaeus aztecus* [brown shrimp]) with a safety factor of 100
Acenaphthene	9.70	USEPA 2001	USEPA Region 4 chronic screening value
Acenaphthylene	6.00	Buchman 2008	Acute LOEL for chemical class with a safety factor of 50 (value for low molecular weight PAHs)
Anthracene	5.35	USEPA 2007	Minimum acute value (48-hr LC_{50} for *Americamysis bahia* [opossum shrimp]) with a safety factor of 100
Benzo(a)anthracene	0.025 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Benzo(a)pyrene	10.0	USEPA 2004	Acute value (LC_{50}) with a safety factor of 100
Benzo(b)fluoranthene	6.00	Buchman 2008	Acute LOEL for chemical class with a safety factor of 50 (value for high molecular weight PAHs)
Benzo(g,h,i)perylene	6.00	Buchman 2008	Acute LOEL for chemical class with a safety factor of 50 (value for high molecular weight PAHs)
Benzo(k)fluoranthene	6.00	Buchman 2008	Acute LOEL for chemical class with a safety factor of 50 (value for high molecular weight PAHs)
Chrysene	10.0	USEPA 2004	Acute value (LC_{50}) with a safety factor of 100
Dibenzo(a,h)anthracene	6.00	Buchman 2008	Acute LOEL for chemical class with a safety factor of 50 (value for high molecular weight PAHs)
Fluoranthene	11.0	USEPA 1996	Final Chronic Value
Fluorene	10.0	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Nereis arenaceodentata* [polychaete]) with a safety factor of 100
Indeno(1,2,3-cd)pyrene	6.00	Buchman 2008	Acute LOEL for chemical class with a safety factor of 50 (value for high molecular weight PAHs)
Naphthalene	23.5	USEPA 2001	USEPA Region 4 chronic screening value
Phenanthrene	8.30	USEPA 1996	Final Chronic Value
Pyrene	0.248	USEPA 2007	Minimum acute value (48-hr LC_{50} for *Americamysis bahia* [opossum shrimp]) with a safety factor of 100
Pesticides (µg/L):			
4,4'-DDD	0.001	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters (value for 4,4'-DDT and metabolites)
4,4'-DDE	0.001	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters (value for 4,4'-DDT and metabolites)
4,4'-DDT	0.001	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters (value for 4,4'-DDT and metabolites)
Aldrin	0.13	USEPA 2001	USEPA Region 4 chronic screening value
alpha-BHC	2.30 [3]	USEPA 2007	Minimum acute value (40-day LC_{50} for *Lymnaea stagnalis* [great pond snail]) with a safety factor of 100
beta-BHC	32.0 [3]	USEPA 2007	Minimum chronic value (12-week NOEC for *Poecilia reticulata* [guppy] based on general histological changes)
Chlordane	0.004	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters

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Chemical	Groundwater Screening Value [1]	Reference	Comment [2]
Pesticides (µg/L) (cont.):			
Chlorobenzilate	0.076	USEPA 2005	Minimum acute value (96-hr NOEC for *Cyprinodon variegatus* [sheepshead minnow]) with a safety factor of 10
delta-BHC	0.125	USEPA 2007	Minimum acute value (96-hour LC_{50} for *Macrobrachium idella idella* [prawn]) with a safety factor of 100
Dieldrin	0.0019	USEPA 2009b	Criteria Continuous Concentration based on Final Residual Value
Endosulfan I	0.0087	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters (value for total endosulfan)
Endosulfan II	0.0087	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters (value for total endosulfan)
Endosulfan sulfate	0.92 [3]	USEPA 2007	Minimum chronic value (21-day LOEC for *Daphnia magna* [cladoceron] based on reproduction) with a safety factor of 10
Endrin	0.0023	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Endrin aldehyde	NA	---	---
gamma-BHC (lindane)	0.16	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Heptachlor	0.0036	USEPA 2009b	Criteria Continuous Concentration based on Final Residual Value
Heptachlor epoxide	0.0036	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Isodrin	0.12 [3]	USEPA 2007	Minimum acute value (24-hr LC_{50} for *Lepomis macrochirus* [bluegill]) with a safety factor of 100
Kepone	0.094	USEPA 2007	Minimum chronic value (28-day MATC for *Americamysis bahia* [opposum shrimp] based on growth)
Methoxychlor	0.03	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Toxaphene	0.0002	PREQB 2010	Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Total Recoverable Metals (µg/L):			
Antimony	500	Buchman 2008	Proposed Criteria Continuous Concentration
Arsenic	36.0	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Barium	16,667	USEPA 2007	Minimum acute value (96-hr NOEC for *Cyprinodon variegatus* [sheepshead minnow]) with a safety factor of 30
Beryllium	167	USEPA 2007	Minimum acute value (96-hr NOEC [NR-ZERO] for *Fundulus heteroclitus* [mummichog]) with a safety factor of 30
Cadmium	8.85	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Chromium (total)	50.4 [4]	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Cobalt	45.0	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Nitocra spinipes* [Harpacticoid copepod]) with a safety factor of 100
Copper	3.73	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Lead	8.52	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Mercury	1.11	USEPA 2009	Total recoverable Criteria Continuous Concentration
Nickel	8.28	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Selenium	71.1	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Silver	2.24	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Thallium	21.3	USEPA 2001	USEPA Region 4 chronic screening value
Tin	180 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Vanadium	12.0 [3]	USEPA 2003	USEPA Region 5 ecological screening level
Zinc	85.6	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Dissolved Metals (µg/L):			
Antimony	500 [5]	Buchman 2008	Proposed Criteria Continuous Concentration
Arsenic	36.0	USEPA 2009	Dissolved Criteria Continuous Concentration for trivalent arsenic
Barium	16,667 [5]	USEPA 2007	Minimum acute value (96-hr NOEC for *Cyprinodon variegatus* [sheepshead minnow]) with a safety factor of 30
Beryllium	167 [5]	USEPA 2007	Minimum acute value (96-hr NOEC [NR-ZERO] for *Fundulus heteroclitus* [mummichog]) with a safety factor of 30
Cadmium	8.80	USEPA 2009	Dissolved Criteria Continuous Concentration
Chromium (total)	50.0	USEPA 2009	Dissolved Criteria Continuous Concentration for hexavalent chromium
Cobalt	45.0 [5]	USEPA 2007	Minimum acute value (96-hr LC_{50} for *Nitocra spinipes* [Harpacticoid copepod]) with a safety factor of 100

APPENDIX A

ECOLOGICAL GROUNDWATER SCREENING VALUES
SWMU 60 - FORMER LANDFILL AT THE MARINA
FULL RCRA FACILITY INVESTIGATION
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Chemical	Groundwater Screening Value [1]	Reference	Comment [2]
Dissolved Metals (µg/L) (cont.):			
Copper	3.10	USEPA 2009	Dissolved Criteria Continuous Concentration
Lead	8.10	USEPA 2009	Dissolved Criteria Continuous Concentration
Mercury	0.94	USEPA 2009	Dissolved Criteria Continuous Concentration
Nickel	8.20	USEPA 2009	Dissolved Criteria Continuous Concentration
Selenium	71.0	USEPA 2009	Dissolved Criteria Continuous Concentration
Silver	2.24 [5]	PREQB 2010	Total recoverable Puerto Rico Water Quality Standard for Class SB coastal and estuarine waters
Thallium	21.3 [5]	USEPA 2001	USEPA Region 4 chronic screening value
Tin	180 [3][5]	USEPA 2003	USEPA Region 5 ecological screening level
Vanadium	12.0 [3][5]	USEPA 2003	USEPA Region 5 ecological screening level
Zinc	81.0	USEPA 2009	Dissolved Criteria Continuous Concentration

Notes:

EC_{50} - Median Effective Concentration
LC_{50} - Median Lethal Concentration
LLPAH - Polynuclear Aromatic Hydrocarbon
LOEL - Lowest Observed Effect Level
MATC - Maximum Acceptable Toxicant Concentration
µg/L - microgram per liter
NA - Not Available
NOEC - No Observed Effect Concentration
NR-ZERO - 0 percent mortality, or 100 percent survival of test organisms
PREQB - Puerto Rico Environmental Quality Board
USEPA - United States Environmental Protection Agency

[1] The values shown are marine/estuarine screening values unless otherwise noted. Estuarine/marine surface water screening values were preferentially used as groundwater screening values since groundwater flow at SWMU 60 toward the E2SS3 estuarine wetland and the Ensenada Honda (saltwater embayment).
[2] The safety factors applied to acute endpoints (i.e., LC_{50}, EC_{50}, NOEC, and LOEL values) and chronic endpoints (i.e., LOELs) are those recommended by Wentsel et al. (1996).
[3] The chemical lacks a marine/estuarine surface water screening value/literature-based toxicity value. The value shown is a freshwater screening value/toxicity value.
[4] The value shown is for hexavalent chromium.
[5] The chemical lacks a screening value expressed as a dissolved concentration. The value shown is based on the total recoverable concentration of the metal in the water column.

Table References:

Buchman, M.F. 2008. NOAA Screening Quick Reference Tables. NOAA OR&R Report 08-1. National Oceanic and Atmospheric Administration, Office of Response and Restoration Division, Seattle, WA.

Puerto Rico Environmental Quality Board (PREQB). 2010. Puerto Rico Water Quality Standards Regulation. Regulation No. 7837. March 31, 2010.

USEPA 2009a. Great Lakes Inititiative Toxicity Data Clearinghouse. http://www.epa.gov/gliclearinghouse/.

United States Environmental Protection Agency (USEPA). 2009. National Recommended Water Quality Criteria. Office of Water and Office of Science and Technology, Washington, D.C. http://www.epa.gov/waterscience/criteria/wqctable/.

USEPA. 2007. ECOTOX User Guide: Ecotoxicology Database System. Version 4.0. http:/www.epa.gov/ecotox/.

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Table References (cont.):

USEPA. 2005. Pesticide Ecological Effects Database: Guidance Manual. Office of Pesticide Programs, Ecological Fate and Effects Division. Database available at: http://www.ipmcenters.org/Ecotox/index.cfm

USEPA. 2003. USEPA Region 5 Ecological Screening Levels Table. http://www.epa.gov/reg5rcra/ca/ESL.pdf.

USEPA. 2001. Region 4 Ecological Risk Assessment Bulletins - Supplement to RQGS. Waste Management Division, Atlanta, GA. http://www.epa.gov/region04/waste/ots/ecolbul.htm.

USEPA. 1996. Ecotox Thresholds. Eco Update, Volume 3, Number 2. Office of Solid Waste and Emergency Response, Washington, D.C. EPA/F-95/038.

Wentsel, R.S., T.W. Pa Point, M. Simini, R.T. Checkai, and D. Ludwig. 1996. Tri-Service Procedural Guidelines for Ecological Risk Assessments. Edgewood Research Development and Engineering Center, Aberdeen Proving Ground, MD. ADA297968.

APPENDIX B
FIELD DATA COLLECTION FORMS

WETLAND DETERMINATION DATA FORM

WETLAND DETERMINATION DATA FORM – Caribbean Islands Region

Project/Site: _____ Municipality/Town: _____ Sampling Date: _____

Applicant/Owner: _____ PR or USVI: _____ Sampling Point: _____

Investigator(s): _____ Ward/Estate: _____

Landform (hillslope, terrace, etc.): _____ Local relief (concave, convex, none): _____ Slope (%): _____

Lat: _____ Long: _____ Datum: _____

Soil Map Unit Name: _____ NWI classification: _____

Are climatic / hydrologic conditions on the site typical for this time of year? Yes _____ No _____ (If no, explain in Remarks.)

Are Vegetation _____, Soil _____, or Hydrology _____ significantly disturbed? Are "Normal Circumstances" present? Yes _____ No _____

Are Vegetation _____, Soil _____, or Hydrology _____ naturally problematic? (If needed, explain any answers in Remarks.)

SUMMARY OF FINDINGS – Attach site map showing sampling point locations, transects, important features, etc.

Hydrophytic Vegetation Present?	Yes _____	No _____	**Is the Sampled Area within a Wetland?** **Yes _____ No _____**
Hydric Soil Present?	Yes _____	No _____	
Wetland Hydrology Present?	Yes _____	No _____	

Remarks:

VEGETATION – Use scientific names of plants.

Tree Stratum (Plot size: _____)	Absolute % Cover	Dominant Species?	Indicator Status
1. _____			
2. _____			
3. _____			
4. _____			
5. _____			
	_____ = Total Cover		
Sapling/Shrub Stratum (Plot size: _____)			
1. _____			
2. _____			
3. _____			
4. _____			
5. _____			
	_____ = Total Cover		
Herb Stratum (Plot size: _____)			
1. _____			
2. _____			
3. _____			
4. _____			
5. _____			
6. _____			
7. _____			
8. _____			
	_____ = Total Cover		
Woody Vine Stratum (Plot size: _____)			
1. _____			
2. _____			
3. _____			
4. _____			
	_____ = Total Cover		

Dominance Test worksheet:

Number of Dominant Species That Are OBL, FACW, or FAC: _____ (A)

Total Number of Dominant Species Across All Strata: _____ (B)

Percent of Dominant Species That Are OBL, FACW, or FAC: _____ (A/B)

Prevalence Index worksheet:

Total % Cover of:		Multiply by:
OBL species	_____	x 1 = _____
FACW species	_____	x 2 = _____
FAC species	_____	x 3 = _____
FACU species	_____	x 4 = _____
UPL species	_____	x 5 = _____
Column Totals:	_____ (A)	_____ (B)

Prevalence Index = B/A = _____

Hydrophytic Vegetation Indicators:

___ Rapid Test for Hydrophytic Vegetation

___ Dominance Test is >50%

___ Prevalence Index is ≤3.0[1]

___ Problematic Hydrophytic Vegetation[1] (Explain)

[1]Indicators of hydric soil and wetland hydrology must be present, unless disturbed or problematic.

Hydrophytic Vegetation Present? **Yes _____ No _____**

Remarks:

SOIL

Sampling Point: _____

Profile Description: (Describe to the depth needed to document the indicator or confirm the absence of indicators.)

Depth (inches)	Matrix Color (moist)	Matrix %	Redox Features Color (moist)	Redox Features %	Type[1]	Loc[2]	Texture	Remarks

[1]Type: C=Concentration, D=Depletion, RM=Reduced Matrix, CS=Covered or Coated Sand Grains. [2]Location: PL=Pore Lining, M=Matrix.

Hydric Soil Indicators:

___ Histosol (A1)
___ Histic Epipedon (A2)
___ Black Histic (A3)
___ Hydrogen Sulfide (A4)
___ Organic Bodies (A6)
___ 5 cm Mucky Mineral (A7)
___ Muck Presence (A8)
___ Depleted Below Dark Surface (A11)
___ Thick Dark Surface (A12)
___ Sandy Gleyed Matrix (S4)
___ Sandy Redox (S5)
___ Stripped Matrix (S6)
___ Dark Surface (S7)
___ Loamy Gleyed Matrix (F2)
___ Depleted Matrix (F3)
___ Redox Dark Surface (F6)
___ Depleted Dark Surface (F7)
___ Redox Depressions (F8)

Indicators for Problematic Hydric Soils[3]:

___ Stratified Layers (A5)
___ Very Shallow Dark Surface (TF12)
___ Other (Explain in Remarks)

[3]Indicators of hydrophytic vegetation and wetland hydrology must be present, unless disturbed or problematic.

Restrictive Layer (if observed):

Type: _____

Depth (inches): _____

Hydric Soil Present? Yes _____ No _____

Remarks:

HYDROLOGY

Wetland Hydrology Indicators:

Primary Indicators (minimum of one required; check all that apply)

___ Surface Water (A1)
___ High Water Table (A2)
___ Saturation (A3)
___ Water Marks (B1)
___ Sediment Deposits (B2)
___ Drift Deposits (B3)
___ Algal Mat or Crust (B4)
___ Iron Deposits (B5)
___ Inundation Visible on Aerial Imagery (B7)
___ Water-Stained Leaves (B9)
___ Aquatic Fauna (B13)
___ Hydrogen Sulfide Odor (C1)
___ Oxidized Rhizospheres on Living Roots (C3)
___ Presence of Reduced Iron (C4)
___ Recent Iron Reduction in Tilled Soils (C6)
___ Thin Muck Surface (C7)
___ Fiddler Crab Burrows (C10)
___ Other (Explain in Remarks)

Secondary Indicators (minimum of two required)

___ Surface Soil Cracks (B6)
___ Sparsely Vegetated Concave Surface (B8)
___ Drainage Patterns (B10)
___ Dry-Season Water Table (C2)
___ Saturation Visible on Aerial Imagery (C9)
___ Geomorphic Position (D2)
___ Shallow Aquitard (D3)
___ FAC-Neutral Test (D5)

Field Observations:

Surface Water Present? Yes _____ No _____ Depth (inches): _____

Water Table Present? Yes _____ No _____ Depth (inches): _____

Saturation Present? (includes capillary fringe) Yes _____ No _____ Depth (inches): _____

Wetland Hydrology Present? Yes _____ No _____

Describe Recorded Data (stream gauge, monitoring well, aerial photos, previous inspections), if available:

Remarks:

DAILY METER CALIBRATION RECORD

DAILY METER CALIBRATION RECORD

Date: _____ Time: _____

Model: YSI 556 MPS
Serial #:

		Initial	Adjusted
pH (Std. Units)			
Buffer:	4		
Buffer:	7		
Buffer:	10		
ORP (mV's)			
Std.:			
Sp.Cond. (mS/cm)			
Std.:			
D.O. (mg/l)			
Baro. Pressure (mm/Hg):			
Temp. (Celsius)			N/A

Model: RAE 2000
Serial #:

	Initial	Adjusted
Isobutylene (100ppm)		

Model:
Serial #:

		Initial	Adjusted
Turbidity (NTU's)			
Std.:	10		

GROUNDWATER SAMPLING FORMS

WELL DETAIL AND SAMPLING LOG

Well ID #: _____ **DATE:** _____

PROJECT: _____ **WEATHER:** _____

WELL DATA

(CONDITIONS): (G = Good, F = Fair, P = Poor)

Pad _____ Well Cap _____ PVC Locking Cap/Plug _____

Cap Lock _____ Cover Bolts _____ Water over PVC? Y / N

Casing (outer) _____ Casing (inner) _____ SWL Reference Mark _____ Reference Mark Location? _____

Casing Dia.: _____ Casing Material: _____ Flushmount / Stickup

Comments: _____

(OTHER): Static Water Level (ft.): _____ Time: _____ (**Pre-Pump** Installation)

Depth to Product (ft.): _____

Water Level (ft.): _____ Time: _____ (Post-Pump Installation, **Pre-Purge**)

Total Well Depth (ft.) - Post Sampling: _____ Time: _____

PID Reading (PPM) - Unopened: _____ Opened: _____

PURGE DATA

Pump Type: _____

From Boring Log: Total Depth (ft.): _____ Screened Interval (ft.): _____

Pump Intake Set @ (ft.): _____ Controller Settings / Pressure: _____

Comments: _____

SAMPLE DATA

Sample ID #: _____ Dup.: Y / N - (ID#: _____)

Sample Time: _____ MS / MSD: Y / N Field Filtered: Y / N

Sampled By: _____ Signature: _____

Sample Description: _____

SAMPLE CONTAINERS

Bottle Type	Qty.	Preservative	Analysis

GENERAL COMMENTS

LOW FLOW PURGE DATA SHEET

WELL ID: _____

DATE: _____ **SAMPLER (s):** _____

PROJECT: _____ **SAMPLE ID:** _____

TIME [3-5 min.]	DEPTH TO WATER (ft) [<0.3 ft.]	VOL. (ml)	PURGE RATE (ml/min) [250-500 ml/min]	TEMP. (°C)	SP. COND. (mS/cm) [+/- 3%]	D.O. (mg/l) [+/- 10%]	pH (S.U.) [+/- 0.1]	ORP (mV) [+/- 10]	TURBIDITY (NTU) [+/- 10%]	COMMENTS (Sample descrip.: color, clarity, odor - issues and adjustments, etc.)
		- (Water Level : Post-Pump Installation, Pre-Start)								
	- (Start of Purging)									
	- **Sample Time** (Note: Flow rate to be btwn. 100 - 250 ml/min.)									**Sampler Signature:**

Note: Stability achieved when 3 consecutive readings fall within specific parameter ranges shown in the above header.

APPENDIX C
USEPA REGION II GROUNDWATER SAMPLING PROCEDURE

U.S. ENVIRONMENTAL PROTECTION AGENCY
REGION II

GROUND WATER SAMPLING PROCEDURE
LOW STRESS (Low Flow) PURGING AND SAMPLING

I. SCOPE & APPLICATION

This Low Stress (or Low-Flow) Purging and Sampling Procedure is the EPA Region II standard method for collecting low stress (low flow) ground water samples from monitoring wells. Low stress Purging and Sampling results in collection of ground water samples from monitoring wells that are representative of ground water conditions in the geological formation. This is accomplished by minimizing stress on the geological formation and minimizing disturbance of sediment that has collected in the well. The procedure applies to monitoring wells that have an inner casing with a diameter of 2.0 inches or greater, and maximum screened intervals of ten feet unless multiple intervals are sampled. The procedure is appropriate for collection of ground water samples that will be analyzed for volatile and semi-volatile organic compounds (VOCs and SVOCs), pesticides, polychlorinated biphenyls (PCBs), metals, and microbiological and other contaminants in association with all EPA programs.

This procedure does not address the collection of light or dense non-aqueous phase liquids (LNAPL or DNAPL) samples, and should be used for aqueous samples only. For sampling NAPLs, the reader is referred to the following EPA publications: DNAPL Site Evaluation (Cohen & Mercer, 1993) and the RCRA Ground-Water Monitoring: Draft Technical Guidance (EPA/530-R-93-001), and references therein.

II. METHOD SUMMARY

The purpose of the low stress purging and sampling procedure is to collect ground water samples from monitoring wells that are representative of ground water conditions in the geological formation. This is accomplished by setting the intake velocity of the sampling pump to a flow rate that limits drawdown inside the well casing.

Sampling at the prescribed (low) flow rate has three primary benefits. First, it minimizes disturbance of sediment in the bottom of the well, thereby producing a sample with low turbidity (i.e., low concentration of suspended particles). Typically, this saves time and analytical costs by eliminating the need for collecting and analyzing an additional filtered sample from the same well. Second, this procedure minimizes aeration of the ground water during sample collection, which improves the sample quality for VOC analysis. Third, in most cases the procedure significantly reduces the volume of ground water purged from a well and the costs associated with its proper treatment and disposal.

III. ADDRESSING POTENTIAL PROBLEMS

Problems that may be encountered using this technique include a) difficulty in sampling wells with insufficient yield; b) failure of one or more key indicator parameters to stabilize; c) cascading of water and/or formation of air bubbles in the tubing; and d) cross-contamination between wells.

Insufficient Yield

Wells with insufficient yield (i.e., low recharge rate of the well) may dewater during purging. Care should be taken to avoid loss of pressure in the tubing line due to dewatering of the well below the level of the pump=s intake. Purging should be interrupted before the water level in the well drops below the top of the pump, as this may induce cascading of the sand pack. Pumping the well dry should therefore be avoided to the extent possible in all cases. Sampling should commence as soon as the volume in the well has recovered sufficiently to allow collection of samples. Alternatively, ground water samples may be obtained with techniques designed for the unsaturated zone, such as lysimeters.

Failure to Stabilize Key Indicator Parameters

If one or more key indicator parameters fails to stabilize after 4 hours, one of four options should be considered: a) continue purging in an attempt to achieve stabilization; b) discontinue

purging, do not collect samples, and document attempts to reach stabilization in the log book; c) discontinue purging, collect samples, and document attempts to reach stabilization in the log book; or d) Secure the well, purge and collect samples the next day (preferred). The key indicator parameter for samples to be analyzed for VOCs is dissolved oxygen. The key indicator parameter for all other samples is turbidity.

Cascading

To prevent cascading and/or air bubble formation in the tubing, care should be taken to ensure that the flow rate is sufficient to maintain pump suction. Minimize the length and diameter of tubing (i.e., 1/4 or 3/8 inch ID) to ensure that the tubing remains filled with ground water during sampling.

Cross-Contamination

To prevent cross-contamination between wells, it is strongly recommended that dedicated, in-place pumps be used. As an alternative, the potential for cross-contamination can be reduced by performing the more thorough Adaily@ decontamination procedures between sampling of each well in addition to the start of each sampling day (see Section VII, below).

Equipment Failure

Adequate equipment should be on-hand so that equipment failures do not adversely impact sampling activities.

IV. PLANNING DOCUMENTATION AND EQUIPMENT

< Approved site-specific Field Sampling Plan/Quality Assurance Project Plan (QAPP). This plan must specify the type of pump and other equipment to be used. The QAPP must also specify the depth to which the pump intake should be lowered in each well. Generally, the target depth will correspond to the mid-point of the most permeable zone in the screened interval. Borehole geologic and geophysical logs can be used to help select the most permeable zone. However, in some cases, other criteria may be used to select the target depth for the pump

intake. In all cases, the target depth must be approved by the EPA hydrogeologist or EPA project scientist.

< Well construction data, location map, field data from last sampling event.

< Polyethylene sheeting.

< Flame Ionization Detector (FID) and Photo Ionization Detector (PID).

< Adjustable rate, positive displacement ground water sampling pump (e.g., centrifugal or bladder pumps constructed of stainless steel or Teflon). A peristaltic pump may only be used for inorganic sample collection.

< Interface probe or equivalent device for determining the presence or absence of NAPL.

< Teflon or Teflon-lined polyethylene tubing to collect samples for organic analysis. Teflon or Teflon-lined polyethylene, PVC, Tygon or polyethylene tubing to collect samples for inorganic analysis. Sufficient tubing of the appropriate material must be available so that each well has dedicated tubing.

< Water level measuring device, minimum 0.01 foot accuracy, (electronic preferred for tracking water level drawdown during all pumping operations).

< Flow measurement supplies (e.g., graduated cylinder and stop watch or in-line flow meter).

< Power source (generator, nitrogen tank, etc.).

< Monitoring instruments for indicator parameters. Eh and dissolved oxygen must be monitored in-line using an instrument with a continuous readout display. Specific conductance, pH, and temperature may be monitored either in-line or using separate probes. A nephalometer is used to measure turbidity.

< Decontamination supplies (see Section VII, below).

< Logbook (see Section VIII, below).

< Sample bottles.

< Sample preservation supplies (as required by the analytical methods).

< Sample tags or labels, chain of custody.

V. SAMPLING PROCEDURES

Pre-Sampling Activities

1. Start at the well known or believed to have the least contaminated ground water and proceed systematically to the well with the most contaminated ground water. Check the well, the lock, and the locking cap for damage or evidence of tampering. Record observations.

2. Lay out sheet of polyethylene for placement of monitoring and sampling equipment.

3. Measure VOCs at the rim of the unopened well with a PID and FID instrument and record the reading in the field log book.

4. Remove well cap.

5. Measure VOCs at the rim of the opened well with a PID and an FID instrument and record the reading in the field log book.
6. If the well casing does not have a reference point (usually a V-cut or indelible mark in the well casing), make one. Note that the reference point should be surveyed for correction of ground water elevations to the mean geodesic datum (MSL).

7. Measure and record the depth to water (to 0.01 ft) in all wells to be sampled prior to purging. Care should be taken to minimize disturbance in the water column and dislodging of any particulate matter attached to the sides or settled at the bottom of the well.

8. If desired, measure and record the depth of any NAPLs using an interface probe. Care should be taken to minimize disturbance of any sediment that has accumulated at the bottom of the well. Record the observations in the log book. If LNAPLs and/or DNAPLs are detected, install the pump at this time, as described in step 9, below. Allow the well to sit for several days between the measurement or sampling of any DNAPLs and the low-stress purging and sampling of the ground water.

Sampling Procedures

9. Install Pump: Slowly lower the pump, safety cable, tubing and electrical lines into the well to the depth specified for that well in the EPA-approved QAPP or a depth otherwise approved by the EPA hydrogeologist or EPA project scientist. The pump intake must be kept at least two (2) feet above the bottom of the well to prevent disturbance and resuspension of any sediment or NAPL present in the bottom of the well. Record the depth to which the pump is lowered.

10. Measure Water Level: Before starting the pump, measure the water level again with the pump in the well. Leave the water level measuring device in the well.

11. Purge Well: Start pumping the well at 200 to 500 milliliters per minute (ml/min). The water level should be monitored approximately every five minutes. Ideally, a steady flow rate should be maintained that results in a stabilized water level (drawdown of 0.3 ft or less). Pumping rates should, if needed, be reduced to the minimum capabilities of the pump to ensure stabilization of the water level. As noted above, care should be taken to maintain pump suction and to avoid entrainment of air in the tubing. Record each adjustment made to the pumping rate and the water level measured immediately after each adjustment.

12. Monitor Indicator Parameters: During purging of the well, monitor and record the field indicator parameters (turbidity, temperature, specific conductance, pH, Eh, and DO)

approximately every five minutes. The well is considered stabilized and ready for sample collection when the indicator parameters have stabilized for three consecutive readings as follows (Puls and Barcelona, 1996):

±0.1 for pH
±3% for specific conductance (conductivity)
±10 mv for redox potential
±10% for DO and turbidity

Dissolved oxygen and turbidity usually require the longest time to achieve stabilization. The pump must not be removed from the well between purging and sampling.

13. Collect Samples: Collect samples at a flow rate between 100 and 250 ml/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 ft. VOC samples must be collected first and directly into sample containers. All sample containers should be filled with minimal turbulence by allowing the ground water to flow from the tubing gently down the inside of the container.

    Ground water samples to be analyzed for volatile organic compounds (VOCs) require pH adjustment. The appropriate EPA Program Guidance should be consulted to determine whether pH adjustment is necessary. If pH adjustment is necessary for VOC sample preservation, the amount of acid to be added to each sample vial prior to sampling should be determined, drop by drop, on a separate and equal volume of water (e.g., 40 ml). Ground water purged from the well prior to sampling can be used for this purpose.

14. Remove Pump and Tubing: After collection of the samples, the tubing, unless permanently installed, must be properly discarded or dedicated to the well for resampling by hanging the tubing inside the well.

15. Measure and record well depth.

16. Close and lock the well.

VI. FIELD QUALITY CONTROL SAMPLES

Quality control samples must be collected to determine if sample collection and handling procedures have adversely affected the quality of the ground water samples. The appropriate EPA Program Guidance should be consulted in preparing the field QC sample requirements of the site-specific QAPP.

All field quality control samples must be prepared exactly as regular investigation samples with regard to sample volume, containers, and preservation. The following quality control samples should be collected during the sampling event:

< Field duplicates
< Trip blanks for VOCs only
< Equipment blank (not necessary if equipment is dedicated to the well)

As noted above, ground water samples should be collected systematically from wells with the lowest level of contamination through to wells with highest level of contamination. The equipment blank should be collected after sampling from the most contaminated well.

VII. DECONTAMINATION

Non-disposable sampling equipment, including the pump and support cable and electrical wires which contact the sample, must be decontaminated thoroughly each day before use (Adaily decon@) and after each well is sampled (Abetween-well decon@). Dedicated, in-place pumps and tubing must be thoroughly decontaminated using Adaily decon@ procedures (see #17, below) prior to their initial use. For centrifugal pumps, it is strongly recommended that non-disposable sampling equipment, including the pump and support cable and electrical wires in contact with the sample, be decontaminated thoroughly each day before use (Adaily decon@).

EPA=s field experience indicates that the life of centrifugal pumps may be extended by removing entrained grit. This also permits inspection and replacement of the cooling water in centrifugal pumps. All non-dedicated sampling equipment (pumps, tubing, etc.)

must be decontaminated after each well is sampled (Abetween-well decon,@ see #18 below).

17. **Daily Decon**

A) Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

B) Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Alconox, for 5 minutes and flush other equipment with fresh detergent solution for 5 minutes. Use the detergent sparingly.

C) Rinse: Operate pump in a deep basin of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

D) Disassemble pump.

E) Wash pump parts: Place the disassembled parts of the pump into a deep basin containing 8 to 10 gallons of non-phosphate detergent solution. Scrub all pump parts with a test tube brush.

F) Rinse pump parts with potable water.

G) Rinse the following pump parts with distilled/ deionized water: inlet screen, the shaft, the suction interconnector, the motor lead assembly, and the stator housing.

H) Place impeller assembly in a large glass beaker and rinse with 1% nitric acid (HNO_3).

I) Rinse impeller assembly with potable water.

J) Place impeller assembly in a large glass bleaker and rinse with isopropanol.

K) Rinse impeller assembly with distilled/deionized water.

18. **Between-Well Decon**

A) Pre-rinse: Operate pump in a deep basin containing 8 to 10 gallons of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.
B) Wash: Operate pump in a deep basin containing 8 to 10 gallons of a non-phosphate detergent solution, such as Alconox, for 5 minutes and flush other equipment with fresh detergent solution for 5 minutes. Use the detergent sparingly.

C) Rinse: Operate pump in a deep basin of potable water for 5 minutes and flush other equipment with potable water for 5 minutes.

D) Final Rinse: Operate pump in a deep basin of distilled/deionized water to pump out 1 to 2 gallons of this final rinse water.

VIII. FIELD LOG BOOK

A field log book must be kept each time ground water monitoring activities are conducted in the field. The field log book should document the following:

< Well identification number and physical condition.
< Well depth, and measurement technique.
< Static water level depth, date, time, and measurement technique.
< Presence and thickness of immiscible liquid layers and detection method.
< Collection method for immiscible liquid layers.
< Pumping rate, drawdown, indicator parameters values, and clock time, at three to five minute intervals; calculate or measure total volume pumped.
< Well sampling sequence and time of sample collection.
< Types of sample bottles used and sample identification numbers.
< Preservatives used.
< Parameters requested for analysis.

- Field observations of sampling event.
- Name of sample collector(s).
- Weather conditions.
- QA/QC data for field instruments.

IX. REFERENCES

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APPENDIX D
COMPUCHEM QUALITY MANUAL AND ANALYTICAL SOPs

QUALITY MANUAL

Quality Manual

CompuChem
A Division Of
Liberty Analytical Corp.

Revision 14
January 25, 2011

501 Madison Ave.
Cary, North Carolina 27513
www.compuchemlabs.com

CompuChem's mission is to work towards a cleaner environment, providing quality analytical services economically and efficiently. Our highly trained staff members work as partners to our clients to support and facilitate environmental solutions.

CompuChem
A Division Of
Liberty Analytical Corp.

Quality Manual for
CompuChem, a Division of Liberty Analytical Corporation

501 Madison Avenue
Cary, NC 27513

Revision **14**

Prepared by:
Judith Dozier
Quality Assurance **Specialist**

Issued Under the Authority of William Waring, President
Effective Date: **January 25, 2011**

William Waring, President

Robert E. Meierer, Vice President and General Manager

James Feldhaus, Quality Assurance Manager

Mark Ross, Vice President of Sales and Marketing

Kenneth Grzybowski, Director, Laboratory Operations,
EPA Project Management, **and LIMS Administrator**

Steven Pruskin, Supervisor, Semivolatiles Laboratory

Cathy Dover, Supervisor, Project Management

Joseph Monn, Supervisor of Wet Chemistry **Laboratory**

Harold Cray, Manager, Information Technology

Edna **Montemayor**, Manager, Accounting

Robert Gates, Manager, Administration

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1.0 Statement of Policy

This Quality Manual summarizes the policies and operational procedures associated with CompuChem, a Division of Liberty Analytical Corporation, in Cary, North Carolina. Specific protocols for sample handling and storage, chain-of-custody, and laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC), Department of Defense Environmental Laboratory Accreditation Program (DoD-ELAP) based on Quality Systems Manual 4.**2** (QSM 4.**2**), ISO 17025, and applicable EPA requirements, regulations, guidance, and technical standards. This manual has been prepared in accordance with the guidance documents listed further in Section 14. Further details on these policies and procedures are contained in SOPs and related documents. This Quality Manual, SOPs, and related documentation describe the quality system for CompuChem. This Quality Manual is not for use with Ohio VAP projects. Please see Ohio VAP specific Quality Assurance Manual.

CompuChem performs chemical analyses for inorganic and organic constituents in water, soil, and biological tissue samples. CompuChem's goal, and primary QA objective, is to produce data that meet customer requirements and are scientifically valid, defensible, and of known and documented quality in accordance with standards adopted by NELAC, DoD-QSM 4.**2**, any applicable state or EPA regulations or requirements, **individual state certifications,** and in accordance with CompuChem's Code of Ethics (Figure 1-1).

CompuChem analyzes Proficiency Test (PT) samples at a minimum of two times per year for each field of proficiency testing following the requirements for PT providers and laboratories in Chapter 2.0 of the NELAC standards. Samples are obtained from a**n** A2LA accredited PT provider. The specific analytes and matrices analyzed are based on the current scope of the laboratory services and accreditations and are documented in a laboratory SOP on PT sample analyses **reports** and in QA PT study files.

CompuChem has been a participant in the Contract Laboratory Program (CLP) since its inception and has established a long-standing rapport with the U.S. Environmental Protection Agency (USEPA) Program Office in Washington, DC, through our CLP Project Officer, and with the Region IV support division office. The EPA administers organic and inorganic Quarterly Blind PT studies for CLP analytes.

The technical and service requirements of all requests to provide analytical services are thoroughly evaluated and summarized by the Vice President and General Manager. Marketing, Customer Services, Project Management, and Laboratory Operations review the summary before commitments are made to accept the work and before the project begins. This includes a review of facilities and instrumentation, current sample load, staffing, and any special method, QC or reporting requirements. All measurements are made using published reference methods or methods developed by CompuChem. Competence with all methods is demonstrated according to the procedure described in Appendix B of this Quality Manual prior to use.

The management of CompuChem is fully and firmly committed to the quality assurance program described in this Quality Manual. The company's policies on ethical practices and its organizational hierarchy ensure that all of its employees are free from undue pressures that may adversely affect the quality of their work. Each director, manager and supervisor, as well as their staff, is assigned, in accordance with this plan, responsibilities and objectives. Key aspects of procedures for sample receiving, chain-of-custody, sample preparation, laboratory analysis, data verification, and reporting are described in this Quality Manual and detailed **in** the laboratory's SOPs. This manual and the SOPs document the elements of CompuChem's QA program.

The QA program is maintained and expanded or modified as necessary, to ensure all reported data are of uncompromising quality. In order to determine whether QA objectives are met, sufficient quality control will be generated. To meet the objectives of the QA program, senior management supports a program designed to:

- assess the capabilities of analytical methods for meeting users' needs in terms of accuracy, precision, completeness, representativeness, and comparability.
- establish and monitor the routine operational performance of the laboratory through appropriate systems checks to ensure that all aspects of the QA program are operative.
- assure that corrective actions are taken and that system control has been restored before resuming sample analysis whenever QC acceptance criteria are not met.

CompuChem has developed a proactive program for prevention of improper, unethical or illegal actions. Components of this program include the following:

- proficiency testing (single blinds)
- electronic data audits and post-analysis data audits by the QA staff
- QA audit of the data management software audit trail for organics analyses
- a signed Code of Ethics statement (Figure 1-1) issued and signed at employee orientation and reaffirmed annually by each employee with their signature
- reaffirmation through an annual review and sign-off by all technical and support staff of the laboratory policies pertaining to manual integrations, instrument data system time, and SOP compliance
- annual supplemental company-wide ethics training
- written SOPs addressing issues such as performing manual peak integrations and proper ethical conduct.

1.1 CODE OF ETHICS

CompuChem, a Division of Liberty Analytical Corporation, provides analytical services to a variety of clients involved in the assessment of toxicants in environment samples. Our commitment to meeting client expectations and providing data of the highest quality has been, and will continue to be, a distinguishing feature of our service. Consistent with the commitment to quality data is the underlying, assumed attribute of integrity as it relates to all aspects of our service business. Because clients put their trust in the testing laboratory's ability to provide impeccable results, CompuChem's policies and philosophy will be never to put that trust in jeopardy.

CompuChem's Standard Operating Procedures (SOPs) provide appropriate instructions for completing the required tasks in the laboratory and support areas. Additional guidance is provided by Good Laboratory Practice (glp) standards, the Quality Manual and appropriate laboratory policy memos. Training programs within each area also supply added assistance that supplements the direction provided by the SOPs. Within the contents of the aforementioned documentation, and in any training programs, provisions are included which govern the situations that allow for changes to written data (including chain-of-custody documentation) or instrument output. Situations requiring change must be appropriately documented and the requirements for this are set forth in the Quality Manual or appropriate SOPs.

In absolutely no circumstances do CompuChem's policies, philosophy, SOPs, or training programs allow for inappropriate manipulation (falsification) within the analytical testing process that would compromise the validity of that process. This practice constitutes fraud and is grounds for termination. CompuChem's management feels very strongly about this position since we value the trust that clients place in the quality and integrity of the data we produce for them.

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Vice President & General Manager

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1.2 Scope of the Quality Assurance Program Outlined in this Manual

The quality assurance plan described in this document applies specifically to CompuChem, a Division of Liberty Analytical Corporation, at 501 Madison Avenue in Cary, North Carolina. The plan pertains to the following major topics:

Policy
Organization and Responsibilities
Quality Assurance Objectives
Sample Handling
Test Methods and Standard Operating Procedures
Reference Standard Preparation and Instrument Calibration
Data Quality
Data Reduction, Review, and Reporting
Performance and System Audits and Frequency
Corrective Action
Facilities, Equipment, Security and Waste Management
Subcontracting and Support Services and Supplies

1.3 Concurrence with Quality Assurance program

The management of CompuChem is committed to the laboratory's quality system and to its continued improvement. The management of CompuChem understands and acknowledges that the laboratory is required to be continually in compliance with the National Environmental Laboratory Accreditation Program (NELAP) Standards and ISO 17025. All of CompuChem's responsible parties document their concurrence with the policies and procedures detailed in this document by their signatures on its Title Page.

1.4 Procedures for Review of Requests, Tenders, and Contracts

Prior to CompuChem entering into a contract to provide analytical testing services, the Vice President of Sales and Marketing reviews the request for bid (tender) and the Request for Proposal (RFP) or Scope of Work (SOW) presented by the client. The RFP or SOW details the technical and service requirements associated with requests to provide analytical services. During, or prior to, the bidding process the Vice President of Sales and Marketing forwards the RFP or SOW to the Vice President and General Manager or the Manager of Quality Assurance, **who thoroughly review the technical aspects of the request,** if non-routine technical or service requirements are requested. After the review has been completed, a written summary is prepared and distributed to Laboratory Operations, Marketing, and Project Management for review. This summary documents the review of the following:

- Verification that the sample container and preservation requirements and the extraction and analyses holding times are consistent with method requirements.
- Verification that the laboratory has the requisite laboratory instrumentation, equipment, and personnel to perform the analytical services.
- Verification that the laboratory performs the analytical methods required for the project.

- Verification that the QC requirements presented are consistent with the method and that the laboratory has the ability to achieve them.

- Verification that the laboratory meets any identified accreditation/certification requirements.

- Verification that **the** laboratory can meet all specific State and/or permit specified methodology, QC criteria, and reporting limit requirements.

- Verification that the laboratory can achieve all method detection limit (MDL) and practical quantitation limit (PQL) requirements.

- Verification that the laboratory is able to meet the hard copy and electronic data deliverable (EDD) requirements.

- The review also identifies any items that need to be resolved with the client. Any differences must be resolved before work is allowed to commence. These may include errors identified during the review of the client-supplied documentation or suggestions on alternative analytical approaches that would meet the project objectives. These observations and suggestions are also documented in the summary. All information pertaining to requests, tenders and contracts are maintained by the laboratory in the applicable project file, the Laboratory Information Management System (LIMS), the intranet server, and the quality assurance department.

- If a contract must be amended after work has commenced, the same contract review process is repeated and all amendments communicated to all affected personnel. Any work which is to be subcontracted must be reviewed by the prospective subcontract laboratory. Work may only be subcontracted to laboratories which posses the requisite accreditation/certification requirements. Subcontracted laboratories must additionally receive project specific approval from the client prior to any samples being analyzed, preferably in writing if at all possible. **In addition, subcontract laboratories are bound by the same ethical standards adopted by CompuChem.**

2.0 Organization and Responsibility

2.1 Introduction

CompuChem offers the scientific and technical expertise needed to service the chemical laboratory needs of our customers. In addition to experienced analytical laboratory personnel who have skills in organic and inorganic methodologies, CompuChem utilizes a computer systems staff that manages software systems for data reporting and sample scheduling and control. To ensure that all the needs of our clients are met, a project manager is assigned to each account, providing a liaison between the customer and the laboratory.

An organizational chart of management personnel is shown in Figure 2-1. This chart includes all individuals discussed further in this section. The Human Resources department maintains job descriptions for all employees. Job descriptions for key technical positions and key management positions shown on the organizational chart are found in Figure 2-**2**. Resumes of key technical and management staff are found in Figure 2-**3**.

This section describes the operational and functional responsibilities of key lab personnel, including the duties and services performed for product quality. The roles and responsibilities of the Quality Assurance Department and its organizational relationship to lab management are identified.

The Quality Assurance staff monitors and reviews all laboratory units and operates independently of production. All quality control criteria are documented and compliance is verified at each level of laboratory data review. Standard Operating Procedures (SOPs) for in-lab data evaluation and independent QA auditing describe the details of these quality control functions. The QA Department is responsible for verifying the integrity of these functions and documenting performance for lab management review.

2.2 Assignment of Responsibilities

The following is a brief summary of the responsibilities and authorities assigned to each of the QA staff, laboratory management staff, and laboratory technical staff:

2.2.1 Vice President and General Manager

Assuring the laboratory achieves all QA Program objectives, the Vice President and General Manager monitors and directs quality activities of QA Department and lab personnel. The Vice President and General Manager acts in strict adherence to the procedures and requirements stated in the Quality Manual. He works closely with laboratory supervisors and staff to address data quality issues. The Vice President and General Manager reports directly to the President. The Vice President and General Manager and QA have oversight responsibilities for all lab operations as depicted in the organization chart. The Vice President and General Manager and QA Manager have the authority to terminate non-conforming work.

The Vice President and General Manager and the QA manager conduct annual assessments of the total QA program. Based on the assessments, a written status report of QA activities and progress is forwarded to management. Additional monthly reports include information regarding the effectiveness of the quality management systems are prepared. Included are:

- status of or changes to the QA program and Quality Manual
- measures of data quality

- significant QA problems, accomplishments and recommendations
- results of performance audits
- results of system audits
- status of QA requirements for contracts and QA Project Plans
- summary of QA training (internal and external QA/QC seminars and courses)
- overall effectiveness of the QA program

2.2.2 Manager of Quality Assurance (*QA Officer*)

The Manager of Quality Assurance reports to the Vice President and General Manager and is organizationally and functionally independent of direct job involvement and day-to-day laboratory operations. The QA Officer serves as the focal point for QA/QC and over**sees** and/or review**s** quality control data **and** is primarily responsible for auditing the implementation of the quality system and carrying out the directives of the Vice President and General Manager.

2.2.3 Director, Laboratory Operations

The **Director,** Laboratory **Operations** reports directly to the President and is responsible for all aspects of laboratory operations. All laboratory section supervisors report directly to the **Director**. **The Director** is responsible for ensuring that all laboratory technical staff are qualified and properly trained to perform their job functions. **The Director** is also responsible for the timely delivery of client data reports that meet all quality control and project requirements.

2.2.4 Laboratory Supervisors

The laboratory section supervisors report directly to the **Director, Laboratory Operations** and are responsible for sample receipt, sample preparation, sample analysis, reference standard preparation, instrument calibration, instrument maintenance, corrective action, data report preparation, and waste disposal.

2.2.5 Laboratory Technical Staff

Laboratory technical staff report directly to section supervisors and are responsible for performing their various job functions utilizing current, approved laboratory standard operating procedures, EPA methods and EPA CLP statements of work. Laboratory technical staff are also responsible for the preparation and analysis of quality control samples and adherence to quality control and safety procedures.

2.2.6 Project Management and Customer Support Services

Project Management representatives are responsible for communicating client and project-specific requirements to the laboratory in order to ensure that data quality objectives are met. The Project Manager is the internal customer representative and the primary contact for the client. Details of the project requirements are reviewed and discussed prior to the receipt of samples. The Laboratory Information Management System (LIMS) allows the Project Manager (PM) to list detailed requirements for a given project. The LIMS is also available to all appropriate personnel.

2.3 Training of Technical Staff

CompuChem's training program is administered by the Human Resources (HR) department, **the QA Department,** and laboratory supervisors, and applies to all full-time, temporary, and part-time employees. Prospective employees must meet all job description requirements in order to be hired. Current employees must meet all job description requirements in order to be promoted. Checklists are used to document that requirements are met. All hiring and subsequent changes in personnel status are documented through the use of the Personnel Action Form (PAF).

Various types of training are provided for employees new to a position, and the training records are maintained in the individual's permanent training files. Initial and on-going performance is measured through indicators such as precision and accuracy in replicate spiked quality system matrices, performance evaluation or single blind proficiency test samples, and surrogate recoveries. Employees who fail to maintain acceptable performance standards must be retrained. Retraining must be documented before the employee may work independently. Training proficiency records are maintained for each employee.

Trainees are not allowed to process samples independently before they have demonstrated capability. See Appendix B of this manual for the policy on performing **a** Demonstration of Capability. During the training period, any work performed by a trainee is directly supervised by a senior qualified analyst, and any worksheets, forms, or other analytical data are reviewed and co-signed by the senior qualified analyst. The training period varies in length of time depending on the position and on the prior experience and knowledge level of the individual. As needed, the QA staff provides training in certain quality aspects of the operation such as correct chain of custody documentation, proper error correction protocols, etc. **A** signatory review of the Quality Manual by all staff documents their familiarization with, and agreement to implement, all policies and procedures in their work.

In order to provide adequate staff to perform all of the laboratory's job functions, analysts are **cross**-trained, typically within their departments, to perform other job functions. These **cross**-trained analysts serve as backup in the absence of the primary analyst, or as additional help, if the work load is heavy in a particular area. **Crossed**-trained analysts are required to demonstrate capability as described in Appendix B in all job functions **for** which they perform.

Each employee is required to read and understand the SOPs relative to their job responsibilities. They must agree to follow the most current version of the controlled SOP. **Signatory e**vidence of this agreement is maintained in each employee's training file or within the QA Department.

All job functions are fully described in formalized job descriptions. The employee job descriptions include minimum acceptable levels of formal education, training, prior experience, and special requirements for certifications or licenses. Some contracts, client agreements, **or external agencies** specify minimum qualifications for technical, administrative, computer, and management positions. Certain positions require auxiliary training, including viewing of training videotapes, on-site training classes, or off-site attendance of specialized training or certification courses.

Formal training at CompuChem also involves safety and chemical hygiene training. The Vice President and General Manager has the ultimate responsibility for chemical hygiene in the laboratory and provides continuing support of the program. The Safety Officer/**Chemical Hygiene Officer (CHO), and** Safety Committee have the responsibility of coordinating and

enforcing the laboratory safety program at CompuChem. Each employee in the laboratory is responsible for ensuring an effective chemical hygiene plan. The safety program involves the following key elements:

- safety training programs for all personnel
- documentation of safety training
- the Chemical Hygiene Plan (CHP), approved by the Vice President and General Manager
- the Emergency Action Plan, approved by the Vice President and General Manager
- monthly inspections of the facilities for compliance with safety regulations
- verification that all safety equipment is operable and in good working condition (including inspection and recharging of all fire extinguishers, and monthly inspection of fume hoods and eye washes)
- initial testing of all new safety equipment
- annual fire/evacuation drills
- the safety committee, which is comprised of safety facilitators representing key sections of the operation
- right-to-know seminars held for all laboratory personnel to discuss chemical hazards, safety precautions, medical treatment, and spill procedures

The CHO and the safety committee are responsible for conducting internal safety inspections, covering all aspects of laboratory safety including fire, hazardous materials, personal dress, electrical safety, posted evacuation routes, and condition of all safety equipment. Laboratory department managers/supervisors are responsible for any corrective actions identified in the inspections or during safety drills.

Laboratory department managers/supervisors are responsible for safety briefings for all employees in their department. Employees receive technical assistance from the CHO in complying with the CHP. Training includes safety for fire, electricity, compressed gases, chemical hazards, safety equipment, and hazardous sample and waste handling, depending on the responsibilities of the department or laboratory. New employees must be trained in all aspects of safety concerned with their job responsibilities and the laboratory in which they work. Human Resources, along with department managers/supervisors and the CHO must maintain documentation of safety training. The documentation must include a completed training form, a list of the attendees, the training subject(s), the time spent in training, and the date.

A variety of local seminars, workshops, and lectures are also made available to employees. Workshop and seminar attendees usually relate their experiences in the form of trip reports or in-house presentations to appropriate staff members.

2.4 Laboratory Capabilities

CompuChem analyzes waste water, soil, and solid and liquid waste samples, and biological tissue. Table 2.1 lists the parameters, analytes, and method references, **while Table 2.2 presents a list of method references**. Unless otherwise indicated in the SOP, the laboratory performs the published method as written.

Figure 2-1:

CompuChem, a Division of Liberty Analytical Corporation
Key Personnel Organizational Chart

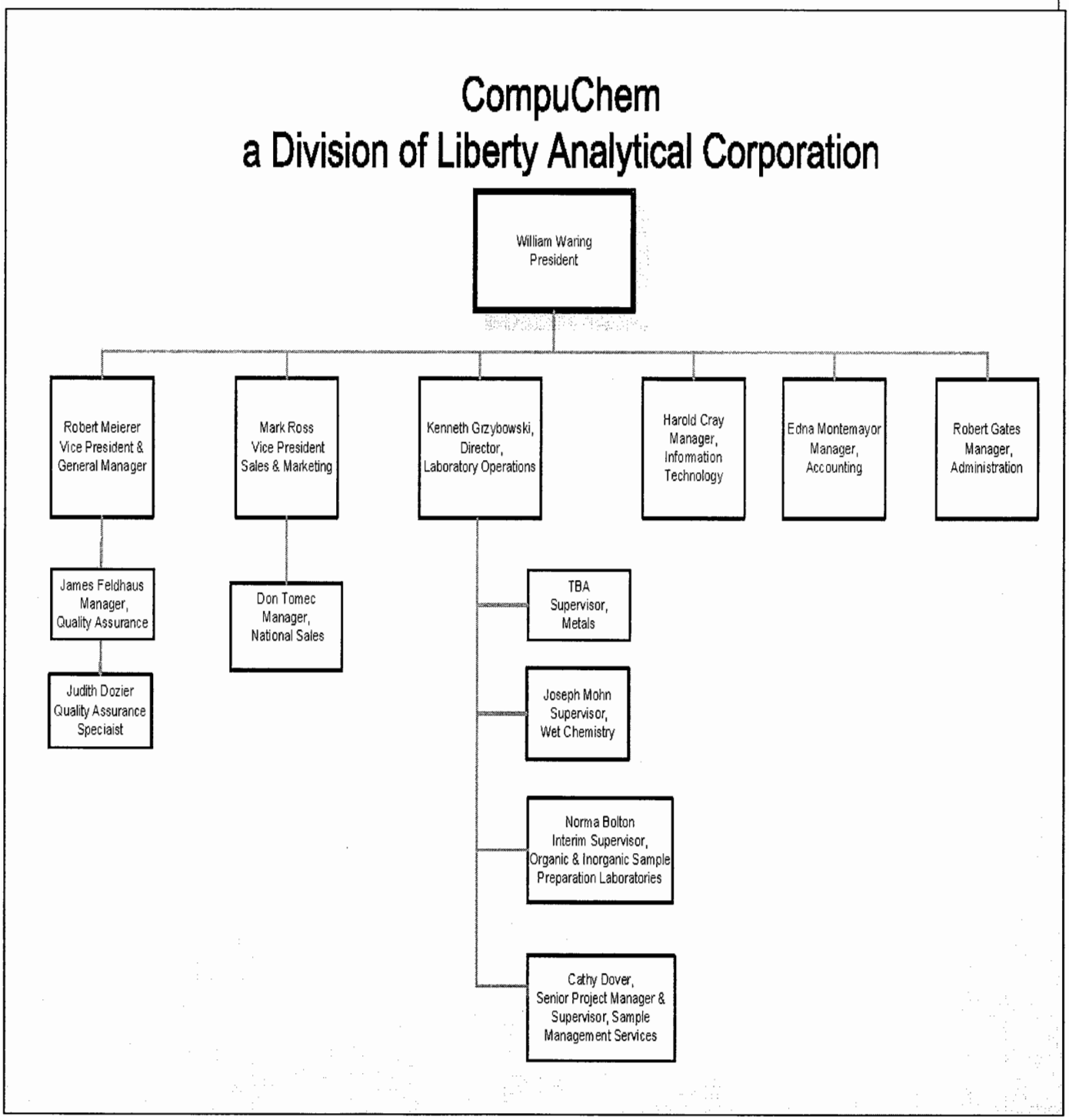

Figure 2-2: ***Job Descriptions of Key Positions listed on Organization Chart***

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Vice President & General Manager*** **JOB NO.**:370008
DEPARTMENT: 3700 – Quality Assurance & Technology **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: President **LOCATION**: Cary, NC

I. BASIC FUNCTION:

Develop and implement strategic processes and practices within the company. Manage and guide the overall organization. Provide guidance in analyzing and appraising the effectiveness of overall operations. Assure all EPA CLP **and DoD** contractual requirements and federal and state agency certification requirements are met by the company. Assure that the company consistently produces data of known and documented quality.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports to and receives direction from the President. This position works closely with **the L**aboratory **Director, Supervisors, Customer Service Representatives,** and the Marketing/Sales personnel concerning the nature and quality of work performed.

Outside of CompuChem, this position works closely with personnel from government agencies on analytical methodologies, customers and potential customers, on the technical aspects of projects, and vendors on project information.

B. Major Functions Reporting to this Position

Quality Assurance Manager

C. Know How, Training and Experience

This position requires an advanced degree in analytical chemistry or equivalent, and a minimum of five (5) years experience in Quality Control/Quality Assurance functions. Extensive management experience and strong management capabilities are required. Experience in EPA and other agency requirements and regulations are required, as is an understanding of government agency interactions. Experience in GC, GC/MS and inorganic analyses **is** also required.

- Plan, develop and manage the Quality Assurance program assuring that all laboratory operations and standards meet the necessary requirements for continuing compliance with accreditation agencies.
- Monitor the effectiveness of the Quality Assurance program, assuring that appropriate QC standards are established and met, implementing changes or new programs as conditions require.
- Resolve quality control problems, explaining or directing the explanation of abnormal sample data, and recommending sample rework or corrective action when necessary.

Vice President & General Manager ***JOB No.: 370008 (continued)***

- Establish the conditions and programs necessary to meet certification and licensing standards on new products/analyses as they are developed.
- Work with laboratories to develop appropriate methodologies and standards for new products or special projects.
- Provide evaluation and approval for all method validation study programs and SOPs.
- Direct in-house systems and performance audits of laboratories and data audits
- Oversee and direct the efforts of the Quality Assurance staff activities to ensure that SOPs and the Quality Manual are current and documented.
- Assess findings in EPA CLP Contract Compliance Screening (CCS) Reports.
- Oversee responses to CCS Reports.
- Assess the feasibility of CLP modified analysis projects requested by the EPA
- Oversee and/or respond to EPA inquiries, data/tape audit reports, and on-site audit reports
- Review available RFP, RFQ and SOW documents for commercial clients and the EPA to understand the analytical and QC requirements
- **Review Proficiency Test (PT) submittal packages and assess any failures, as warranted.**

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Vice Pres. – Sales/Marketing*** **JOB NO.**: 730001
DEPARTMENT: 7300 - Sales & Marketing **F.L.S.A.** E-17
IMMEDIATE SUPERVISOR: President **LOCATION:** Cary

I. BASIC FUNCTION:

Responsible for development of new business (sales) from the industrial, governmental and architect/engineering sectors, and increasing established base business from existing traditional markets/accounts. The position is national in scope, however, specific markets (i.e., CLP) and national areas/regions may be given primary focus (e.g., Atlanta, New York, New Jersey) in terms of sales penetration/concentration. The position will also be responsible for assisting in the selection, training and management of additional staff. This position is responsible for representing the company at Trade Seminars, Conventions, Sales Shows, etc., as may be required to maintain a positive, viable and highly visible corporate image in the market place.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports directly to the President. A vital role is the interface between client and laboratory, specifically, lab operations, customer service and project management personnel in the development of customer specific plans.

B. Major Functions Reporting to this Position

National and Local Sales Representatives

C. Know How, Training and Experience

This position requires a Bachelor of Science in Business Administration, or Science, or equivalent, and prior customer service/sales experience in a technical environment. A demonstrated ability to deal effectively with the public via telephone and in personal face-to-face meetings is absolutely essential as is a sound knowledge of laboratory and general organizational procedures. Excellent interpersonal skills are a must as are strong communication skills and meticulous attention to detail. A positive attitude, proactive problem solving ability and good judgment are absolute requirements given the requirement of the position to effectively manage multiple activities. Efficient time management skills are highly necessary in the performance of this position.

III. ACCOUNTABILITIES:

- Make contact with target audience and qualify prospects, sell and close new and existing customer accounts.
- Provide monthly Sales Forecasts, identifying sold and awarded work anticipated for receipt, "possibles" (awarded work) which may come in and "could-be's" (i.e., projects with submitted proposal not yet awarded) for distribution to management.
- **Discuss and prepare of Annual Sales and Marketing Business and Strategy Plans and formulate new product lines, market penetrations, directions, etc.**

Vice Pres. – Sales/Marketing **JOB NO.**: 730001 (continued)

- Attend Executive Staff Meetings with reports on activities and new business when not traveling.
- Assist Customer Service in getting set-up information gathered and entered accurately, and facilitating generation of customer accounts for review and approval of client and laboratory operations personnel.
- Develop and monitor a successful in-house telemarketing program that compliments direct outside sales efforts by providing established, qualified leads for expert sales attention and closure.
- Maintain awareness of all Federal, State, and local rules and regulations that pertain to employment practices, i.e., wage & hour laws, EEO, and other OSHA regulations.
- Participate in special development projects as required.
- Forecasting - The Territory Overview Report is an essential tool for laboratory operations and is necessary in determining staffing needs (i.e., work schedules/rotations for lab personnel) based on work which is anticipated for receipt on a weekly basis. Control of sample receipts at the back door in an accurate manner is essential to the success of the laboratory. Forecasts by Week, Month and Quarter are required to the extent possible. This will require frequent contact with clients in order to determine where they are with plans for mobilization and sampling on events which have either been awarded outright, or tentatively so, to the company.
- Sales Quotations - Sales quotations, when issued, will be the responsibility of the incumbent. Without exception, any quotation issued which results in an award must be forwarded by fax or otherwise, with appropriate account set-up information to Customer Service prior to shipment of glassware to the client's site.
- Proposals - Submissions necessitate bid package integration requiring the company's communal resources of sales/marketing, customer service, project management, QA, and laboratory personnel. Submissions which require this effort should be date stamped, when received, with a copy forwarded to management for distribution and review ASAP. Ideally, the package should be brought in for review with staff at Executive or Operations Meetings in order that assignments for review.
- Office Attendance - Travel, if planned properly should take place for the most part between Monday and Friday.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Director, Laboratory Operations*** **JOB NO.: 500009**
DEPARTMENT: 5000 Lab Support **F.L.S.A. E-17**
IMMEDIATE SUPERVISOR: President **LOCATION:** Cary, NC

I. BASIC FUNCTION:

Direct the overall day-to-day activities of the GC/MS, GC, Inorganics/Wet Chem. **and** Extractions Laboratories, which are comprised of various technical departments **that** function in a multi-shift production environment. The major responsibility of **the Director of** Laboratory **Operations** is to produce accurate and timely data packages based on the analyses of environmental samples. Additionally, a major responsibility is to develop new methods and/or techniques **that** enhance CompuChem's product line and/or improves its process techniques and production through-put. Further duties include monitoring standards of performance in quality control and quality assurance, and monitoring the validity of the analyses performed and data generated in the laboratory to assure reliable data.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports to and receives direction from the President. This position works closely with the Laboratory Technical Managers, as well as Managers from other CompuChem departments, such as Sales & Marketing, Management Information System, Accounting, and Quality Assurance .

Outside of CompuChem, contact is with CompuChem's customers concerning laboratory practices, status of analyses, explanation of analytical results, etc. This position also has contact with equipment manufacturers, vendors, and consultants utilized by CompuChem.

B. Major Functions Reporting to this Position

Volatile and Semi-Volatile GC/MS Laboratories – Responsible for the preparation and analysis of environmental samples and for the generation of complete data packages.

Extractions Laboratory – Responsible for the preparation of sample extracts for analysis by other laboratories.

GC/HPLC Laboratory – Responsible for the analysis of environmental samples and for the generation of complete data packages.

Inorganics/Wet Chemistry Laboratory – Responsible for the preparation and analysis of inorganics samples, preparation of samples for metals, mercury, cyanide, phenols, fluoride, sulfate, chloride, and other wet chemistry parameters for the generation of complete data packages.

C. Know How, Training and Experience

The position requires a Bachelor's Degree in the chemical, environmental, biological, or physical sciences, or engineering, with at least 24 college credit hours in chemistry and/or at least two years experience in the environmental analysis of representative inorganic and organic analytes for which the laboratory maintains accreditation. A Master's or doctoral degree in one of the above disciplines may be substituted for one year of experience.

Director, Laboratory Operations **JOB NO.: 500009 (continued)**

III. ACCOUNTABILITIES:

- Responsible for directing the Extractions, GC, HPLC, GC/MS, and Inorganics/Wet Chem. Laboratories in the preparation, analysis, and generation of technical data packages of environmental samples within prescribed quality limits and specified timeframes in order to meet production requirements for shipments.
- Direct the development and implementation of new methods/processing improvements in order to allow CompuChem to expand its product line and/or to maximize instrument/processing capability and efficiency. The new methods/processing improvements may deal with analytical procedures, software enhancements or process flow improvements.
- Responsible for the preparation of the annual budget in order to accurately anticipate the cost associated with producing our products and to provide a framework for managing and controlling departmental resources and expenses.
- Develop annual goals and objectives within Laboratory Operations in order to provide a methodical systematic approach to planning and achieving business plan objectives.
- The position will have Profit & Loss (P&L) responsibility for the operation of the laboratory and will be charged with exercising control over various laboratory cost centers in order to accentuate efficiency and the use of purchased materials, reduce expenses to the extent possible, and maximize company profit.
- Responsibility for managing direct reports in the area of P&L responsibility and for assisting in the development of individual supervisors for accepting accountability for controlling costs in their respective lab areas.
- Responsible for ensuring that all pertinent information gleaned in executive staff meetings is disseminated to direct reports in order that communication may be provided on a real-time basis, openly and accurately.
- Participate in special assignments/projects as required.

IV. MAJOR CHALLENGES OF THIS POSITION:

- The most difficult and/or complex part of this position is to integrate a production orientation into a scientific environment which is highly dynamic due to the growth of the business. In combination with the previous statement, the blending of multiple technologies, i.e., analytical chemistry, computers, production, and management expertise to increase the quality of the total organic data package (i.e., reduce reworks of all types) and simultaneously increase productivity is also extremely complex.
- The position will be directly responsible for meeting monthly production quotas in order that shipments may be made in order to allow company to meet monthly revenue and profit goals.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Manager, Purchasing, Admin., Facilities & Chemical Hygiene Officer*** **JOB NO.**: 600005
DEPARTMENT: 6000 Gen. Management **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: *President* **LOCATION**: Cary, NC

I. *BASIC FUNCTION*

Responsible for the purchase of materials, supplies, etc. for all the departments.

Manage facility related activities, including building systems maintenance, telephone system installation and maintenance, and installation of CRT wiring; develop and implement facility renovation plans to provide proper work areas for both office, laboratories, manage hazardous waste collection and disposal program and company wide safety program; design, build and coordinate laboratory support equipment; coordinate cafeteria and vending machine service with contractor.

II. SCOPE OF THE POSITION

A. Organizational Relationships

This position reports to and receives direction from the President. This position works closely with managers/supervisors and all other employees across the Company concerning purchasing, facility and safety issues. The position also serves as contact for CompuChem with vendors for facilities-related projects, telecommunications and Government Regulatory Agencies.

B. Major Functions Reporting to This Position

Building Maintenance

C. Know How, Training and Experience

This position requires a minimum of five (5) years experience in facilities and instrumentation management, including the design and construction of new and existing office and laboratory facilities as well as coordinating and scheduling work with outside contractors and the installation and service of laboratory instruments. Must have knowledge of plumbing system, electrical systems, HVAC systems, telecommunications systems and services, CRT systems, security and fire alarm systems, and Halon systems equipment repair practices. The ability to read and develop blueprints is required. An understanding of safety practices and regulations is required. Knowledge of hazardous waste disposal requirements is also required.

III. ACCOUNTABILITIES

- Operate and maintain laboratory and office heating, ventilating, air conditioning systems and fume hoods. Also responsible for installation and renovation of new and existing systems to meet facility requirements.
- Operate and maintain laboratory and office electrical systems. Also responsible for installation and renovation of new and existing systems to meet facility requirements.
- Operate and maintain plumbing and water systems and the installation and renovation of new and existing systems to meet facilities requirements.

Manager, Purchasing, Admin., Facilities & Chemical Hygiene Officer ***JOB NO.: 600005 (continued)***

- Operate and maintain telecommunications equipment. Audit monthly telecommunications invoices for accuracy and approve for payment. Maintain state-of-the-art telecommunications equipment and services.
- Maintain laboratory appliances.
- Responsible for hiring, training and development of maintenance staff.
- Operate and maintain facility fire suppression systems and fire alarm equipment.
- Manage the facilities shop, assuring that parts and supplies are ordered in a cost-effective manner and making changes in suppliers and/or equipment as required.
- Responsible for the design and construction of new and existing space as required to meet facility needs.
- Work with the laboratory operations management on the adaptations and/or development of special equipment or projects as needed.
- Operation and maintenance of building security systems and personnel.
- Design, fabricate and/or coordinate laboratory support apparatus and equipment.
- Coordinate operations of cafeteria and food vending services.
- Install laboratory gas supply systems.
- Responsible for accounts payable inquiries and remittances.
- Assist in accounts receivable inquiries.
- Responsible for the purchase of all materials & supplies.
- Provide recommendation for promotion and lateral transfers. Conduct performance appraisals, recommend and review merit increases.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Manager of Quality Assurance*** **JOB NO.**: 370002
DEPARTMENT: 3700- Quality Assurance & Technology **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: Vice President & General Manager **LOCATION**: Cary, NC

I. BASIC FUNCTION:

Manage and improve the laboratory's quality assurance program by conducting internal systems audits and coordinating corrective actions with laboratory management and staff.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports to and receives direction from the Vice President and General Manager in matters relating to data quality.

Outside of CompuChem, contact is with State and Federal Regulatory agencies and clients.

B. Major Functions Reporting to this Position

Quality Assurance Specialist

C. Know How, Training and Experience

This position requires an undergraduate degree in Chemistry or related science or equivalent and a minimum of five (5) years laboratory experience, preferably in organic/analytical/clinical chemistry and five (5) years involvement in QA/QC.

III. ACCOUNTABILITIES:

- Develop and evaluate methods for evaluating the quality of data being generated to ensure method and contract compliance.
- Represent the laboratory and the Quality Assurance Department, when required, during on-site audits and laboratory evaluations.
- Perform data and system audit functions and report findings to laboratory management.
- Oversee and participate in the requirements for State Certification programs and PE sample programs.
- Coordinate/perform subcontractor lab audits and provide recommendations for approval of such labs, if required.
- Maintain QA/QC databases, including control limits, MDL and reporting limits, and Corrective Action Reports (CARs).
- Review client QA Project Plans, contracts and statement-of-work, and provide other supplemental Marketing Department support as needed.
- Review and approve Standard Operating Procedures and Method Validation Study data.
- Perform special project functions at the direction of the Vice President and General Manager

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Supervisor, Inorganics*** **JOB NO.:** 430001
DEPARTMENT: 4300 Inorganics **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: Director, Laboratory Operations **LOCATION:** Cary, NC

I. BASIC FUNCTION:

Responsible for the preparation and analysis of various types of environmental samples utilizing instrumental and wet-chemistry procedures for the generation of complete data packages, ensuring that timely and accurate production is achieved. Responsible for analysis of environmental inorganic samples utilizing ICP/**AES**, ICP/MS, Cold Vapor Atomic Absorption spectroscopy, and the generation and transfer of complete data in an accurate and timely manner.

II. SCOPE OF THE POSITION:

A. Organizational Relationships:

This position reports to and receives direction from the **Director,** Laboratory **Operations**. This position works closely with other **supervisors** for the purpose of coordinating production and special projects.

Outside of CompuChem, contact is with vendors, equipment manufacturers, and consultants utilized by CompuChem. This position also provides technical guidance to customers concerning analyses performed and/or participates in on-site audits.

B. Major Functions Reporting to this Position:

None

C. Know How, Training and Experience

This position requires an undergraduate degree in chemistry or equivalent. A minimum of three (3) years of experience in applied analytical techniques (including ICP) for Inorganic Environmental analysis is required. Must have three years of supervisory experience with a strong production orientation.

III. ACCOUNTABILITIES:

- Responsible for ensuring the analytical quality of all data generated by the Inorganic Laboratory.
- Evaluate and develop methods for improving the quality and quantity of the data produced.
- Develop and monitor all training programs utilized in the Inorganic Laboratory for effectiveness and compliance with existing analytical quality standards.
- Plan and schedule work assignments according to analysis requirements. Assign individual work schedules based on analysis requirements and capabilities of the department staff.
- Responsible for interviewing, selecting, orienting, and training new employees.
- Determine training needs of current employees and define a plan of action to address the training requirements.
- **Provide recommendations for promotions and lateral transfers. Conduct performance appraisals, recommend merit increases and review merit increases with employees.**
- **Responsible for communicating and ensuring that all departmental employees understand and adhere to all company policies and procedures.**
- **Responsible for safety attitudes and practices; and for the overall housekeeping of the Inorganic Laboratory.**

Supervisor, Inorganics ***JOB NO.: 430001 (continued)***

- Maintain an accurate inventory of laboratory supplies and equipment. Reorder supplies when required and maintain appropriate records of purchase. Keep expenses to a minimum.
- Responsible for maintaining up-to-date Standard Operating Procedures for the Inorganic Laboratory.
- Assist other areas of the lab by providing personnel and cross-training whenever possible.
- Provide technical guidance and input as requested for new contract requirements, special, and/or new products.
- Ensure overtime is evenly distributed between staff and kept to a minimum.
- Maintain an awareness of all Federal, State, & local rules and regulations that pertain to employment practices, i.e., wage & hour laws, EEO and OSHA regulations
- Responsible for monitoring the absence and vacation utilization by departmental staff.
- Responsible for communicating & ensuring that all departmental employees understand & adhere to all Company's Work Rules; and Policies & Procedures.
- Participate in special assignments/projects as required.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Supervisor, Wet Chemistry*** **JOB NO.**: 440002
DEPARTMENT: 4400 Wet Chemistry **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: Director, Laboratory Operations **LOCATION:** Cary, NC

I. BASIC FUNCTION:

Responsible for the analysis of various types of environmental samples utilizing Total Organic Carbon, **Ion Chromatography**, Colorimetric Automated Analyzers and wet-chemistry procedures for the generation of complete data packages ensuring that timely and accurate production **are** achieved.

II. SCOPE OF THE POSITION:

A. Organizational Relationships:

This position reports to and receives direction from the **Director,** Laboratory **Operations**. This position works closely with other **supervisors** for the purpose of coordinating production and special projects.

Outside of CompuChem, contact is with vendors, equipment manufacturers, and consultants utilized by CompuChem. This position also provides technical guidance to customers concerning analyses performed and/or participates in on-site audits.

B. Major Functions Reporting to this Position:

None

C. Know How, Training and Experience

This position requires an undergraduate degree in chemistry or equivalent. A minimum of three (3) years of experience in applied analytical techniques for Wet Chemistry Environmental analysis is required.

III. ACCOUNTABILITIES:

- Responsible for ensuring the analytical quality of all data generated by the Wet Chemistry Laboratory.
- Evaluate and develop methods for improving the quality and quantity of the data produced.
- Develop and monitor all training programs utilized in the Wet Chemistry Laboratory for effectiveness and compliance with existing analytical quality standards.
- Plan and schedule work assignments according to analysis requirements. Assign individual work schedules based on analysis requirements and capabilities of the department staff.
- Responsible for interviewing, selecting, orienting, and training new employees.
- Determine training needs of current employees and define a plan of action to address the training requirements.

Supervisor, Wet Chemistry Lab ***JOB NO.: 440002 (continued)***

- Provide recommendations for promotions and lateral transfers. Conduct performance appraisals, recommend merit increases and review merit increases with employees.
- Responsible for communicating and ensuring that all departmental employees understand and adhere to all company policies and procedures.
- Responsible for safety attitudes and practices; and for the overall housekeeping of the Wet Chemistry Laboratory.
- Maintain an accurate inventory of laboratory supplies and equipment. Reorder supplies when required and maintain appropriate records of purchase. Keep expenses to a minimum.
- Responsible for maintaining up-to-date Standard Operating Procedures for the Wet Chemistry Laboratory.
- Assist other areas of the lab by providing personnel and cross-training whenever possible.
- Provide technical guidance and input as requested for new contract requirements, special, and/or new products.
- Ensure overtime is evenly distributed between staff and kept to a minimum.
- Maintain an awareness of all Federal, State, & local rules and regulations that pertain to employment practices, i.e., wage & hour laws, EEO and OSHA regulations
- Responsible for monitoring the absence and vacation utilization by departmental staff.
- Responsible for communicating & ensuring that all departmental employees understand & adhere to all Company's Work Rules; and Policies & Procedures.
- Participate in special assignments/projects as required.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Senior Project Manager*** **JOB NO.**: 750010
DEPARTMENT: 7500 Lab Support **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: **Director, Laboratory Operations** **LOCATION**: Cary, NC

I. BASIC FUNCTION:

Manage client projects and provide customer service.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports to and receives direction from the **Director, Laboratory Operations**. The position will work closely with **other** members of Project Management**'s** personnel**,** in order to facilitate timely log-in of customer sample receipts. The position will interface daily with all laboratory areas and personnel relative to the distribution of samples for analysis. The position will work **to** ensure that subcontracted work is documented on a real-time basis and to insure performance of vendor network.

B. Major Functions Reporting to this Position

Project Managers

C. Know How, Training and Experience

The position requires a Bachelor's Degree or a minimum of 5-7 years experience involving the processing and scheduling of receipts in a production environment. Prior supervisory experience is also required as is a working knowledge of LIMS. An understanding of extractions and/or analytical instrumentation processes would be useful.

III. ACCOUNTABILITIES:

- Supervise and train personnel in the areas of responsibility, establish daily workload priorities on a six day schedule and assist in and direct the completion of the activities. Responsible for scheduling personnel and their work time to address and meet workload requirements.
- Responsible for interaction with subcontract **laboratories,** as required**,** in order to schedule shipments for all subcontracted parameters.
- Responsible for updating (as needed) and maintaining as current, all Standard Operating Procedures (SOPs) pertaining to the operation of the area. Responsible for having all personnel familiar with and adhering to them at all times.
- Responsible for preparation of glassware shipments via cooler or bulk distribution to clients as required by client. The position will ensure that clients receive appropriate bottleware, preservatives (if required), COCs, labels, etc. to ensure success of field sampling events.
- Responsible for maintaining a safe, healthy, clean and presentable work environment at all times.
- Maintain awareness of all Federal, State, and local rules and regulations that pertain to employment practices, i.e., wage and hour laws, EEO, and OSHA regulations.
- Responsible for communicating and ensuring that all departmental employees understand and adhere to all Company's Work Rules, and Policies and Procedures.
- Assist other areas of the lab by providing personnel and cross-training whenever possible.

Senior Project Manager ***JOB NO.:*** **750010** ***(continued)***

- Ensure compliance with client Scope of Work (SOW).
- Answer**, or oversee response to,** any technical inquiries from client.
- Sign outgoing reports.
- Gather information from client on project specific requirements.
- Communicate all issues and concerns with lab.
- Review project status and compliance.
- Prepare status reports and inquiry responses as required by client.
- Monitor financial compliance with client agreement.
- Ensure client is satisfied with lab.
- The position requires weekend work and overtime as may be required.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Supervisor, Sample Preparation Lab*** **JOB NO.**: 360001
DEPARTMENT: 3600 Sample Preparation Lab **F.L.S.A.** Exempt
IMMEDIATE SUPERVISOR: **Director, Laboratory Operations** **LOCATION**: Cary, NC

I. BASIC FUNCTION:

Preparation of various environmental matrices using liquid-liquid and liquid-solid chemical extraction techniques for analysis using SW-846, CLP, **and other published** methodologies. Supervising and training of sample preparation technicians and ensuring that timely and accurate production is achieved. Handling and coordinating the disposal of hazardous waste generated by the facility.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports to and receives direction from the **Director,** Lab**oratory Operations**. This position works closely with the other supervisors and managers to coordinate sample processing throughout the laboratory.

B. Major Functions Reporting to this Position

Glassware Preparation – preparation and inventory of all glassware utilized by the various laboratory functions.

Organic Sample Preparation Technicians/analysts – preparation of samples for organic analyses.

C. Know How, Training and Experience

This position requires an undergraduate degree in chemistry or equivalent. A minimum of three (3) years in preparation of environmental samples**, and** one (1) year supervisory experience**.**

III. ACCOUNTABILITIES:

- Ensuring that the production of the sample preparation laboratory is conducted in a timely and accurate manner.
- Plan and schedule work assignments according to sample load.
- Assign individual work schedules based on sample load and capabilities of the department staff.
- Interview, select, and train new employees. Determine training needs of employees and define a plan of action to address the training requirements.
- Provide recommendation for promotions and lateral transfers. Conduct performance appraisals, recommend merit increases and review merit increases with employees.
- Practice overall good housekeeping of the sample and glassware preparation areas.
- Maintain an accurate inventory of laboratory supplies and equipment. Reorder supplies when required and maintain appropriate records of purchase.
- Maintain up-to-date Standard Operating Procedures for the preparation lab
- Monitor absence and vacation utilization by departmental staff.
- Participate in special development projects as required.

COMPUCHEM, a division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Supervisor, Sample Management Services*** **JOB NO.**: 500006
DEPARTMENT: 7500 **Customer Service/Project Management** **F.L.S.A. Non-**Exempt
IMMEDIATE SUPERVISOR: Director, Laboratory Operations **LOCATION**: Cary, NC

I. BASIC FUNCTION:

Supervise the daily activities in the Sample Receiving/Storage and Shipping Areas. Supervise and perform sample receipt, sample-login and order entry. Provide customer service.

II. SCOPE OF THE POSITION:

A. Organizational Relationships

This position reports to and receives direction from the Laboratory Manager. The position will work closely with **other** members of Project Management**'s** personnel, in order to facilitate timely log-in of customer sample receipts. The position will interface daily with all laboratory areas and personnel relative to the distribution of samples for analysis. The position will work closely with Subcontract Coordinator to insure that subcontracted work is documented on a real-time basis, and to insure performance of vendor network.

Outside CompuChem, the position will communicate with shipping vendors to coordinate pick-ups, glassware shipment logistics, etc. This position will also communicate directly with clients as required to resolve issues related to sample receipt, log-in and/or order-entry.

B. Major Functions Reporting to this Position

Sample Custodian

C. Know How, Training and Experience

The position requires a Bachelor's Degree or a minimum of 5-7 years experience involving the processing and scheduling of receipts in a production environment. Prior supervisory experience is also required as is a working knowledge of LIMS. An understanding of extractions and/or analytical instrumentation processes would be useful.

III. ACCOUNTABILITIES:

- Supervise and train personnel in the areas of responsibility, establish daily workload priorities on a six day schedule and assist in and direct the completion of the activities. Responsible for scheduling personnel and their work time to address and meet workload requirements.
- Responsible for the completeness and correctness of all Chain of Custody documentation, including; ensuring proper COC procedures are followed for samples being received and internal COC guidelines are maintained.
- Responsible for identification of discrepancies between sample receipts and client COC documentation and for insuring that appropriate feedback is given clients in order to affect and facilitate timely corrections prior to entering client receipts into the LIMS. Responsible for ensuring those clients receive acknowledgment of sample receipt within 24 hours of order-entry.
- **Responsible for scheduling shipments for all subcontracted parameters.**
- **Responsible for updating (as needed) and maintaining as current, all Standard Operating Procedures (SOPs) pertaining to the operation of the area. Responsible for having all personnel familiar with and adhering to them at all times.**

Supervisor, Sample Management Services ***JOB NO.:*** ***500005 (continued)***

- Responsible for preparation of glassware shipments via cooler or bulk distribution to clients as required by client. The position will ensure that clients receive appropriate bottleware, preservatives (if required), COCs, labels, etc. to ensure success of field sampling events.
- Responsible for maintaining the organization of walk-in and extract coolers daily and for ensuring that all sample request sheets received from each laboratory area are responded to completely, accurately, and in a timely manner with all signature requirements. Ensure sample security at all times by maintaining internal COC.
- Responsible for the copy center, including the photocopying, scanning, and mailing of data packages
- Responsible for maintaining a safe, healthy, clean and presentable work environment at all times.
- Responsible for the hiring, training and development, and performance evaluations of employees in the department. Responsible for documenting sub-par work related performance with employees as may be necessary and for dealing with work related disciplinary problems on an as needed basis. Provide recommendation for promotions, lateral transfer, and merit increases.
- Responsible for maintaining temperature checks of thermometers for short and long-term storage areas and rotate sample stock to **affect** the SOP required purging process on a timely basis.
- Oversight of the purging process after samples have exceeded their retention times.
- Monitor inventory levels of warehouse stock items, glassware, coolers, components (COCs, labels, etc.) to ensure that appropriate levels are maintained on an ongoing basis.
- Maintain awareness of all Federal, State, and local rules and regulations that pertain to employment practices, i.e., wage and hour laws, EEO, and OSHA regulations.
- Responsible for communicating and ensuring that all departmental employees understand and adhere to all Company's Work Rules, and Policies and Procedures.
- Assist other areas of the lab by providing personnel and cross-training whenever possible.
- The position requires weekend work and overtime as may be required.

COMPUCHEM, a Division of Liberty Analytical Corporation
DESCRIPTIVE TITLE: ***Manager Information Technology*** **JOB NO.: 430007**
DEPARTMENT: 4070 Computer Operations **F.L.S.A. Exempt**
IMMEDIATE SUPERVISOR: President **LOCATION: Cary, NC**

I. BASIC FUNCTION:

Manage the computer operations function in support of Company's production, administration, and system management. **Provide technical expertise (hardware configurations, software revisions, user profiles, backup, and recovery) for system use and program development.**

II. SCOPE OF THE POSITION:

A. Organizational Relationships:

This position reports to and receives direction from the President. The position also interacts with any other user within the Company that relies on computerized production systems. This position works closely with the managers and supervisors throughout CompuChem and WearCheck USA.

Outside of CompuChem, the position has contact with hardware vendors and consultants, as well as hardware and software maintenance personnel.

B. Major Functions Reporting to this Position

Computer Support Specialist

C. Know How, Training and Experience

This position requires a minimum of B.S. degree in Computer Science or equivalent and 6 years of experience in computer operations management, a thorough understanding of integrated computer systems including computer maintenance. Also, effective human relation skills are also extremely important in managing and developing department personnel as well as interacting with the various users of the computer systems.

III. ACCOUNTABILITIES:

- Manage the Company's Computer Operations functions, assuring a fully functioning computer system adequate to support the Company's needs.
- Evaluate and select computer hardware and related products.
- Propose hardware configuration including migration/growth strategies.
- Participate in the selection of computer software and related products.
- Manage and control software installations, revisions, configuration and documentation.
- Manage system account structure setup.

Manager, Information Technology ***JOB NO.: 430007 (continued)***

- Ensure that system backup procedures are being followed and that a disaster plan exists.
- Oversee the installation, maintenance and monitoring of computer hardware including mainframes, printers, disk, modems, plotters, and personal computers, as well as the software and programs run on the hardware.
- Interview, select, and train new employees; provide recommendations for promotions and lateral transfers; conduct appraisals and recommend merit increases.
- Provide planned needs of all projects within the computer operations area.
- Maintain up-to-date Standard Operating Procedures for the department.
- Prepare job descriptions for each position or job title within the department. Ensure that all employees have a current job description in their training file. Ensure the written job descriptions have complete signatures including employee, immediate supervisor and next managerial level.
- Ensure that all departmental employees understand and adhere to all Company Policies and Procedures.
- Participate in special development projects are required.

Figure 2-3 Resumes of Key Laboratory Personnel and Technical Directors

Norma Bolton
Interim Supervisor, Sample Preparation Laboratories

Responsibilities: Ms. Bolton is responsible for supervising and training of preparation technicians. Ms. Bolton's responsibilities also include preparation of various environmental matrices using liquid-liquid and liquid solid extraction techniques, use of various cleanup techniques to further prepare the resulting extracts for analysis by SW-846, CLP and other published methodologies.

Education: Ms. Bolton received a GED from Wake Technical Community College in 1984.

Experience: Previously Ms. Bolton was responsible for the preparation (digestion) of various environmental matrices for Cold Vapor and Spectrometer analysis. Her responsibilities include analysis of the resulting digestates using the Cold Vapor technique, along with various other Wet Chemistry techniques. Prior to moving to the Inorganics lab, Ms. Bolton was employed in the organic sample preparation lab from 1990 to 2007.

Ms. Bolton joined CompuChem in February 1987 in the Forensic Drug Testing division as Clinical Laboratory Assistant. She was employed by that division until 1990 in the positions of Senior Clinical Laboratory Assistant, Clinical Sample Preparation Technician Trainee, and Clinical Sample Preparation Technician.

Seniority Date: February 24, 1987

Dover, Cathy
Supervisor of Project Management
Supervisor of Sample Management Services

Responsibilities: Ms. Dover is responsible for representing the laboratory as the primary contact with commercial clients, account setup and maintenance, tracking the status of projects within the production laboratory and providing technical evaluation and support. Ms. Dover ensures that client requirements are met and that proper customer service and technical support are provided during the course of projects.

Ms. Dover is also responsible for overseeing the daily operations of the Sample Management Services department. This department is responsible for receiving samples, providing daily assistance to Project Management in logging samples into the LIMS following their receipt, coordinating and shipping proper sampling media and supplies to clients, copying data for shipment to clients, providing services as Sample Custodians, disposing of date residual samples and the archiving, retrieval, and storage of scanned customer data.

Education: Ms. Dover earned diploma from Charles E. Jordan High School in Durham County in June 1980. She continued her studies at North Carolina State University where she focused on the curriculum in the schools of Chemical Engineering, Chemistry and Science Education.

Experience: Ms. Dover joined CompuChem in August 1983 as a Senior Laboratory Assistant. She earned her first promotion in February 1985 to Quality Control and Report Integration Clerk.

In January 1986, Ms. Dover became Laboratory Production Coordinator, and in August1989 she worked in the position of Production Scheduler/Planner.

In January 1994, as Account Administration Representative, she worked closely with clients on a service level. In April 1997, Ms. Dover was promoted to Special Project Coordinator (CELDM) and dedicated her efforts providing customer service to one of our most significant clients.

In September 1998 Ms. Dover was promoted to the position of Supervisor of Sample Control. In that capacity Ms. Dover was responsible for sample management, including receipt of samples, sample custody, storage and control, and shipping collection materials to the field.

In May 2005 Ms. Dover was promoted from Senior Project Manager to Supervisor of Project Management.

Seniority Date: August 15, 1983

Feldhaus, James C.
Quality Assurance Manager

Responsibilities: **Mr. Feldhaus is responsible for developing and maintaining the laboratory's quality system through the performance of internal systems and data audits and oversight of Proficiency Test sample analyses. He is also responsible for maintaining the laboratory's accreditations with state and federal agencies and the National Environmental Laboratory Accreditation Program (NELAP).**

Education: Mr. Feldhaus received his Bachelor of Science degree in Chemistry from North Carolina State University in 2000.

He has earned 51 credit hours in chemistry.

Experience: **Before being promoted to Quality Assurance Manager in August of 2010,** Mr. Feldhaus **was responsible for overseeing the daily operations of the inorganics department. This included sample preparation, sample analysis, verifying that quality control criteria are met, and the generation of client reports.**

Mr. Feldhaus began his career at CompuChem in May of 2005 as an Analyst III. In that position his responsibilities were the analyses of environmental matrices using ICP-AES and ICP-MS instrumentation, and review of the data produced from those analyses.

Prior to joining CompuChem, Mr. Feldhaus was employed as a Chemist by Chemical and Environmental Technology, Inc in Cary, North Carolina from 2000 to 2005. His responsibilities included environmental sample collection, analysis of environmental matrices using GC, Cold Vapor, ICP-MS, and classical chemistry techniques.

Mr. Feldhaus meets the requirements in Section 4.1.1.1 of NELAC to serve as a Technical Director.

Seniority Date: May 2, 2005

Gates, Robert (Rob) J.
Administrative Manager

Responsibilities: Mr. Gates is Administrative Manager for CompuChem. This position manages all purchasing functions. Mr. Gates also manages all facility related activities, including building systems maintenance. Mr. Gates also serves as the Safety Officer/**Chemical Hygiene Officer**.

Education: Mr. Gates received a B.S. degree in Public Recreation from Georgia Southern University in 1975.

Experience: Before joining CompuChem, Mr. Gates was the Health Physics Supervisor for Alara Engineer. Mr. Gates started this position in June 1992 where he tracked, scheduled, and audited health physics monitoring. From 1986 to 1992 Mr. Gates held the same position for Site Services at the Savannah River site.

Seniority Date: July 8, 1996

Grzybowski, Kenneth
Director, Laboratory Operations

Responsibilities: **Mr. Grzybowski is responsible for managing the laboratory operations and supervising all the departmental personnel in their various capacities. He is also instrumental in the Element LIMS process and provides directional oversight to the IT Department in determining its priorities. Grzybowski also provides data/case review, when time permits, and serves as EPA Project Manager.**

Education: Mr. Grzybowski earned a Bachelor of Science degree in Environmental Earth Science from Buffalo State College in Buffalo, New York.

Experience: **Mr. Grzybowski was named Laboratory Operations Manager in March of 2009 after being Co-Operations Manager since April of 2008. In October 2009, he returned to his former position in the Volatiles lab. In September 2010, he was once again named as Director, Laboratory Operations.**

Prior to rejoining CompuChem, Mr. Grzybowski was employed by Laboratory Corporation of America, Durham, North Carolina as a Senior Technician. His responsibilities included analysis of biological samples for drugs of abuse utilizing GC/MS instrumentation and the troubleshooting and maintenance of that instrumentation.

As VOA Mgr, Mr. Grzybowski was primary responsible for the analysis of environmental samples for volatile constituents utilizing GC and GC/MS instrumentation and the generation of complete and accurate data reports associated with these analyses. He also ensured that the data reported meets quality control and client requirements.

From April 2002 to September 2005, Mr. Grzybowski was employed by CompuChem as an Analyst II. His responsibilities included the analysis of environmental samples for volatile organic compounds using GCMS instrumentation. He ensured the instrument met all calibration and quality control requirements.

From December 1998 to April 2002, Mr. Grzybowski was employed at Severn Trent Laboratories in Amherst, New York, where he held the positions of Volatile Organic Chemist and Laboratory Waste Disposal Manager.

Seniority Date: April 15, 2002

Mohn, Joseph J.
Supervisor, Wet Chemistry

Responsibilities: Mr. Mohn is responsible for the daily operations of the Wet Chemistry Laboratory. This includes sample preparation, sample analysis, verifying that quality control criteria are met, and the generation of client reports.

Education: Mr. Mohn completed his AAS degree in the Environmental Science Technology at Wake Technical Community College in July 2004.

Experience: Prior to being promoted to Wet Chemistry Supervisor in October, 2008, Mr. Mohn held the position of Analyst I. In that position, his responsibilities included the preparation and analyses of environmental matrices using classical wet chemistry techniques.

Prior to joining CompuChem May 2001, Mr. Mohn was employed by Wake County Public School System. From 1998 to 1999, Mr. Mohn was employed by Dogwood Lane Farm as Farm Manager.

Seniority Date: May 30, 2001

Meierer, Robert (Bob) E.
Vice President & General Manager

Responsibilities: Develop and implement strategic processes and practices within the company. Manage and guide the overall organization. Provide guidance in analyzing and appraising the effectiveness of overall operations. Assure all EPA CLP contractual requirements and federal and state agency certification requirements are met by the company. Assure that the company consistently produces data of known and documented quality.

Education: Mr. Meierer received an Associate degree in Industrial Chemistry from the Erie County Technical Institute in 1963, and an undergraduate B.A. degree in Chemistry from the State University of New York at Buffalo in 1971. He has taken advanced studies in Analytical Chemistry and Business Administration from the State University at Buffalo.

During his studies Mr. Meierer earned 76 credit hours in chemistry toward his degree. No credit hours in microbiology were required for his degree program.

Experience: As Vice President of Quality Assurance and Technology at CompuChem Mr. Meierer was responsible for assuring that the laboratory consistently produced high quality and reliable data, and that all necessary certification and licensing requirements were met by the laboratory. Additional responsibilities included the implementation of change, defined by new technology or operational improvement needs.

Prior to joining CompuChem, Mr. Meierer held positions as Laboratory Manager with Radian Corporation and as Department Head, Analytical Laboratory; Special Contamination Monitoring, The Carborundum Company from 1969-1980.

In his previous position with CompuChem Corporation as Vice President of Quality, Mr. Meierer was responsible for overseeing the QA efforts for both the environmental analytical and forensic drug testing aspects of the company.

Through the variety of laboratory positions Mr. Meierer has held, he has gained fifteen (15) years of experience in the interpretation of GC/MS volatile and semivolatile data. In addition, Mr. Meierer has six (6) years of experience in the preparation of extracts from environmental or hazardous waste samples.

Further, he has gained ten (10) years of experience in organochlorine pesticide residue and PCB analysis, including clean-up procedures (such as column chromatography) for a variety of environmental sample matrices.

Mr. Meierer meets the requirements in Section 4.1.1.1 of NELAC to serve as a Technical Director.

Meierer, Robert (Bob) E.
Vice President & General Manager

Publications:

Meierer, R. E., "Non-Traditional Analyses on CLP Contracts." paper presented at the 19th Annual National Environmental Monitoring Conference, Crystal City Hilton, Arlington, VA, on July 23, 2003.

Meierer, R. E., "The Analytical Testing Laboratory: Its Role and Responsibility in Environmental Legislation." paper presented at the Environmental Science for Lawyers Seminar, NC Bar Center, Cary, NC, on February 10, 1995.

Meierer, R.E., "How to Maintain a High Level of Quality Assurance--and Still Make a Profit," paper presented at the Third Annual Conference of the International Association of Environmental Testing Laboratories, Virginia Beach, VA, on October 23, 1990.

Meierer, R.E., Whitehead, R.J. "Making an On-Site Evaluation of an Analytical Services Laboratory," Environmental Claims Journal, Vol. 1, 4 (Summer 1989)

L.R. Flynn, S.W. Bass, R.E. Meierer, "Headspace Screening/Capillary Column GC/MS Analysis for Volatile Organics: Validation Studies and Applications." paper presented at the Fifth Annual Waste Testing and Quality Assurance Symposium, Washington, DC, on July 24-28, 1989.

Meierer, R.E., Whitehead, R.J. "Quality Assurance: In Search of Excellence," Environmental Lab, November 1989.

Meierer, R.E., "Selecting and Evaluating An Analytical Service Laboratory: What the Client Should Do and What the Responsibilities of the Laboratory Are." paper presented at the Environmental-Analytical Teleconference on Quality, on November 10, 1988.

Meierer, R.E., Ragsdale, P.L. "Validation of Toxicity Characteristic Leaching Procedure (TCLP) and Application to Industrial Wastes," paper presented in Washington, DC at the USEPA Symposium on Solid Waste Testing and Quality Assurance, July 13, 1987.

Meierer, R.E., Myers R.L., Whitehead, R.J., "Quality Assurance Studies Based On Analytical Condition Codes," paper presented to the Fifth Annual EPA Contract Laboratory Program Conference, U.S. EPA, August 1, 1985.

Meierer, R.E., "GC/MS: Applications For The Determination of Organic Constituents In Hazardous Waste," paper presented at the Twelfth Annual Conference on Waste Technology, NSWMA, October 18, 1983.

Meierer, Robert (Bob) E.
Vice President & General Manager

Publications:

Meierer, R.E., Ragsdale P.L., and Mills, P.E., "Quality Assurance of Support Functions In A Large Hazardous Wastes Analytical Laboratory," paper presented before the division of Environmental Chemistry, American Chemical Society, March 29, 1982.

Meierer, R.E., "Laboratory Data Credibility and Reliability," paper presented in Milwaukee, Wisconsin on March 8, 1980, at the Federation of Environmental Technologists Conference.

Shaffer, P.T.B., Meierer, R.E., McGee, C.D., "Virus Recovery From Natural Water" JAWWA., 69 (10), 528-531 (1977).

Cook, G.A., Meierer, R.E., and Shields, B.M. "Combustibility Tests on Several Flame-Resistant Fabrics in Compressed Air, Oxygen Enriched Air, and Pure Oxygen." Textile Research, 37:591 (1967).

Seniority Date: October 2, 1980

Ross, Mark S.
Vice-President Sales and Marketing

Responsibilities: Mr. Ross is responsible for the marketing and sales of services among major Federal programs and commercial clients. He is responsible for directing the efforts of local and national sales staff. He also continues to maintain his involvement in the Waste Management Program, and is familiar with all regulations pertaining to transportation and handling of hazardous waste.

Education: Mr. Ross earned an AAS degree in Medical Technology from Biscayne Paramedical Institute (Miami, FL) in 1976. He also attended Otterbein College (Wasterville, OH) for two years and has completed various professional development courses in effective management, communication, chemical safety and storage, and atomic spectroscopy.

Experience: Before joining CompuChem, Mr. Ross was employed as Inorganic Analytical Chemistry Manager at Battelle (Columbus, OH) from April 1988 to February 1991. From February 1987 until March 1988, he was Inorganic and Water Chemistry Supervisor at Radian Corporation (Sacramento, CA), and from May 1984 to December 1986, he was a Senior Research Technician at Battelle. From December 1977 to May 1984, Mr. Ross was a Senior Research Technician at Owens Corning Fiberglass Technical Center (Granville, OH).

Mr. Ross joined CompuChem as an Inorganic Off-Shift Supervisor in February 1991, and was promoted to Inorganic Production Manager in November 1991. In October 1994 Mr. Ross expanded his duties to include waste management.

In November 1996 Mr. Ross was promoted to his current position.

Seniority Date: February 4, 1991

Table 2.1 - Analytical Methods Performed by CompuChem

Method ID	Type of Analysis	Analyte	Method References
Clean Water Act Methods			
130.1	Colorimetric, flow Injection analyzer, Lachat	total hardness as CaCO3	EPA, 1971 Lachat, 2/20/01
4500-H^+B	Electrometric	pH	Standard Method, 20th Ed. 1998
2540C	Gravimetric	filterable residue, total dissolved solids (TDS)	Standard Methods 20th Ed. 1998
2540D	Gravimetric	non-filterable residue, total suspended solids(TSS)	Standard Methods 20th Ed. 1998
2540B	Gravimetric	total solids	Standard Methods 20th Ed. 1998
300.0	Ion Chromatography	Bromide	EPA/6006R-93/100 EPA, Rev. 2.1, 1993
300.0	Ion Chromatography	Chloride	EPA/6006R-93/100 EPA, Rev. 2.1, 1993
300.0	Ion Chromatography Distillation	Fluoride	EPA/6006R-93/100 Std.Meth. 20th Ed. EPA, Rev. 2.1, 1993
300.0 353.2	Ion Chromatography Colorimetric, flow Injection analyzer, Lachat	nitrate N	EPA/6006R-93/100 EPA, Rev. 2.1, 1993
300.0 353.2	Ion Chromatography Colorimetric, flow injection analyzer, Lachat	nitrite N	EPA/6006R-93/100 EPA, Rev. 2.1, 1993 EPA, Rev. 2.0, 1993
300.0	Ion Chromatography	orthophosphate P	EPA/6006R-93/100 EPA, Rev. 2.1, 1993
300.0	Ion Chromatography	Sulfate	EPA/6006R-93/100 EPA, Rev. 2.1, 1993
310.2	Colorimetric, flow Injection analyzer, Lachat	Alkalinity	EPA, 1974 Lachat, January 2001
335.4 4500 Cn-**I** 10-204-00-1-A	Off-line manual distillation, colorimetric, flow injection analyzer, Lachat	Cyanide, total and free Cyanide, amenable	EPA, Rev. 1.0, 1993 Standards Methods 20th Ed. 1998 Lachat, March 1992
350.1 **10-107-06-1-A**	**Colorimetric** **flow injection analyzer,** **Lachat distillation**	**Ammonia N**	**EPA, Rev.2, 1993** **Lachat, November 1995**
351.2 **10-107-06-2-E**	**Colorimetric,** **flow injection analyzer,** **Lachat**	**Kjeldahl N, total**	**EPA, Rev. 2.1, 1993** **Lachat, June 1996**
3500-Fe D/B	**Phenanthroline method**	**Ferrous/ferric iron**	**Std.Meth. 19th/20th Ed.**

Table 2.1 - (cont.) Analytical Methods Performed by CompuChem

Clean Water Act Methods (cont.)

365.4 10-115-01-1-D	colorimetric, flow injection analyzer, Lachat	total phosphorus P	EPA, 1974 Lachat, May 1995
4500-S^2-F	titrimetric	Sulfide	Standard Methods 20th Ed. 1998
1664A	gravimetric	Hexane-extractable material, HEM	EPA-821-R-98-002
1664A	gravimetric	Silica-gel treated (SGT)-HEM	EPA-821-R-98-002
5310B	DC-180 total organic carbon analyzer	TOC	Standard Methods 20th Ed. 1998
420.4 10-210-00-1-A	manual distillation, automated colorimetric, 4-AAP, Lachat	Phenols	EPA, Rev. 1.0, 1993 Lachat, April 1987
3030C	aqueous filtration/acid digestion	Metals	Std.Meth. 20th Ed.
10-124-13-1-A 3500-CrD/B	automated colorimetric, flow injection analyzer, Lachat	Hexavalent chromium (Cr+) (Aqueous)	Lachat, October 1993 Std.Meth. 19th/20th Ed.
D3987-85	Leachate generation from soil	Wet chemistry Parameter	ASTM 1999
RSK-175	GC/FID, gas in water (headspace) analysis	Methane, ethane, ethene, propane	RSK-175

Table 2.1 - (cont.) Analytical Methods Performed by CompuChem

Resource Conservation & Recovery Act Methods

Method ID	Type of Analysis	Analytes	Method References
1010**A** ASTM-D93-**80**	Pensky-Martens closed cup	ignitability	EPA SW846, 3rd Ed., **Up. IIIB, 11/04**
1311	toxicity characteristic leaching procedure (TCLP)	semivolatiles, metals, herbicides, pesticides	EPA SW846, 3rd Ed., Up.I, 7/92
1311	TCLP w/ zero headspace extraction (ZHE)	volatile organics	EPA SW846, 3rd Ed., Up.I, 7/92
D3987-85	leachate generation from soil	Wet chemistry (S)	ASTM
1312	synthetic precipitation leaching procedure (SPLP)	semivolatiles, metals, herbicides, pesticides dissolved metals	EPA SW846, 3rd Ed., Up.II, 9/94 3rd Ed., Up.I, 7/92
3010A	aqueous acid block-digestion ICP	total metals (Aqueous)	EPA SW846, 3rd Ed., Up.I, 7/92
3050B	soil/sediment/sludge acid block-digestion	total metals (Solids)	EPA SW846, 3rd Ed., Up.III, 12/96
3510C	separatory funnel liquid/liquid extraction	organic extractables (Aqueous)	EPA SW846, 3rd Ed., Up.III, 12/96
3520C	continuous liquid/liquid extraction	organic extractables (Aqueous)	EPA SW846, 3rd Ed., Up.III, 12/96
3535**A**	solid phase extraction (SPE)	Explosives (Aqueous)	EPA SW846 3rd Ed., Up.III, 2/**07**
3540C	soxhlet extraction	extractable organics (Solids)	EPA SW846, 3rd Ed., Up.III, 12/96
3541	Automated Soxhlet Extraction (SoxTherm)	Semivolatiles & PCBs (Solids)	EPA SW846, 3rd Ed., Up.III,12/96
3550B, 3550C	sonication extraction	non-volatile & extractable organics (Solids)	EPA SW846, 3rd Ed., Up.III, 12/96 Up. IV (2/07)
3580A	waste dilution, dilute & shoot	non-aqueous waste	EPA SW846, 3rd Ed., Up.I, 7/92
3620B	florisil column cleanup	Organochlorine pesticides & PCBs	EPA SW846, 3rd Ed., Up.III, 12/96
3640A	gel permeation cleanup (GPC)	extractable organics	EPA SW846, 3rd Ed., Up.II, 9/94
3660B	sulfur cleanup	organochlorine pesticides & PCBs	EPA SW846, 3rd Ed., Up.III, 12/96
3665A	sulfuric acid cleanup	PCBs	EPA SW846, 3rd Ed., Up.III, 12/96
3005A	**Aqueous acid block digestion**	**Total recoverable or dissolved metals**	**EPA SW846, 3rd Ed., Up.I, 7/92**
5030B	**P&T**	**volatile organics**	**EPA SW846, 3rd Ed., Up.III, 12/96**

Table 2.1- (cont.) Analytical Methods Performed by CompuChem

Method ID	Type of Analysis	Analytes	Method References

Resource Conservation & Recovery Act Methods (cont.)

5035, 5035A(draft)	Closed system P&T for soil	volatile organics	EPA SW846, 3rd Ed., Up.III, 12/96, 7/07
6010B, 6010C	ICP	metals	EPA SW846, 3rd Ed., Up.III, 12/96 Up. IV (2/07)
6020, 6020A	ICP-MS	metals	EPA SW846, 3rd Ed.,Up. III, 12/96 Up. IV (2/07)
7196A	automated colorimetric, flow injection analyzer, Lachat	Hexavalent chromium (Cr+) (Aqueous)	EPA SW846, 3rd Ed.,Up. III, 12/96
7470A, 7471A, 7471B	CVAA automated Leeman manual cold-vapor	Mercury	EPA SW846, 3rd Ed., Up. II, 9/94, Up. IV (2/07)
8015B, 8015C	GC/FID	Gasoline range organics, including TN-DEC Diesel range organics, including JP4, JP8,TN EPH (Aqueous & Solids)	EPA SW846, 3rd Ed., Up.III, 12/96 Up. IV (2/07)
8081A, 8081B	solvent extraction, GC/ECD	Organochlorine Pesticides	EPA SW846, 3rd Ed., Up.III, 12/96 Up. IV (2/07)
8082, 8082A	solvent extraction, GC/ECD	PCBs	EPA SW846, 3rd Ed., Up.III, 12/96 Up. IV (2/07)
8151A	solvent extraction, GC/ECD	chlorinated herbicides	EPA SW846, 3rd Ed., Up.III, 12/96
8260B	GC/MS P&T Selected ion monitoring (SIM)	purgeable volatile Organics	EPA SW846, 3rd Ed., Up.III, 12/96
8270C, 8270D	solvent extraction, GC/MS capillary column Selected ion monitoring (SIM)	semivolatile organic Extractables	EPA SW846, 3rd Ed., Up.III, 12/96 Up. IV (2/07)
8310	solvent extraction, HPLC/UV/fluorescence	polynuclear aromatic hydrocarbons (PAHs)	EPA SW846, 3rd Ed., 9/86
8330, 8330A, 8330B	solvent extraction; Solid and Aqueous (8330B – Aqueous only) HPLC,UV detector	Explosives	EPA SW846, 3rd Ed., Up.II, 9/94 Up. IV (2/07)
8332	solvent extraction, HPLC,UV detector	Nitroglycerin/PETN	EPA SW846, 3rd Ed., Up. III, 12/96
9010B, **9010C**	manual midi-distillation (followed by 9012A/**9012B**, flow injection analysis,Lachat)	total and free cyanide	EPA SW846, 3rd Ed., Up. III, 12/96, **3rd Ed., Up. IIIB, 11/04**

Table 2.1 - (cont.) Analytical Methods Performed by CompuChem

Resource Conservation & Recovery Act Methods (cont.)

Method ID	Type of Analysis	Analytes	Method References
9012A, **9012B**	off-line midi-distillation, flow injection analyzer, Lachat	total and free cyanide	EPA SW846, 3rd Ed., Up.III, 12/96, **3rd Ed., Up. IIIB, 11/04**
Chapter (7.3.3) 9014	HCN formation and release, titration	reactive (total releasable) cyanide	EPA SW846, 3rd Ed., Up.III, 12/96
Chapter (7.3.4) 9034	H2S formation and release, titration	reactive (total releasable) sulfide	EPA SW846, 3rd Ed., 9/86
9040B	electrometric	aqueous pH	EPA SW846, 3rd Ed., 1/95
9045C, **9045D**	electrometric	soil & waste pH (corrosivity)	EPA SW846, 3rd Ed., Up.IIB, 1/95, **3rd Ed., Up. IIIB, 11/04**
9060A, modified for soil/water Lloyd Kahn	DC-180 TOC analyzer	TOC (Aqueous & Solids)	EPA SW846, 3rd Ed., **11/04**
9066	manual off-line distillation, automated colorimetric, flow injection analyzer, Lachat	Phenol (Solids)	EPA SW846, 3rd Ed., 9/86, CE-81-1, 5/81
9070A (see Method 1664)	gravimetric	Oil and Grease (Aqueous)	EPA SW846, 3rd Ed.,Up.III, 12/96
9071B	soxhlet extraction, gravimetric Hexane-Extractable Material (HEM)	Oil and Grease (Solids)	EPA SW846, 3rd Ed., Up.III, 12/96 Rev.2, April 1998
9095A	filtration	paint filter liquids	EPA SW846, 3rd Ed., Up.III, 12/96

Table 2.1 - (cont.) Analytical Methods Performed by CompuChem

Superfund / Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), EPA Contract Laboratory Program (CLP)

Method ID	Type of Analysis	Analytes	Method References
EPA CLP	ICP/AES	inorganic metals, total & dissolved	CLP SOW **ILM05.4**
EPA CLP	ICP/MS	Inorganic metals, total & dissolved	CLP SOW ILM05.4
EPA CLP	CVAA	Mercury	CLP SOW **ILM05.4**
EPA CLP	manual distillation, flow injection analyzer, Lachat	Cyanide total	CLP SOW **ILM05.4**
EPA CLP	GC/MS P&T **megabore column** **Full-scan and SIM**	Trace & low-medium aqueous volatiles Low/**medium** solid volatiles	CLP SOW SOM01.2
EPA CLP	solvent extraction, GC/MS capillary column **Full-scan and SIM**	semivolatile organic aqueous Low/**medium** solid	CLP SOW SOM01.2
EPA CLP	solvent extraction, GC/ECD capillary column	organochlorine pesticides	CLP SOW SOM01.2
EPA CLP	solvent extraction, GC/ECD capillary column	Aroclors	CLP SOW SOM01.2

Table 2.2: Method References

Method Key	**Reference**
ASTM	American Society for Testing and Materials, D3987-85 (1999), Standard Test Method for Shake Extraction of Solid Waste with Water; D93-**80**, Standard Test Method for Flash-Point by Pensky-Martens Closed Cup Tester
40 CFR 136	Code of Federal Regulations 40, Part 122, 136 (March 12, 2007) "Guidelines Establishing Test Procedures for Analysis of Pollutants Under the Clean Water Act."
40 CFR 136, Appendix A	Appendix A. (since October 26, 1984, with subsequent updates.) "Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater."
40 CFR **136**, Appendix C	Appendix C. (since October 26, 1984, with subsequent updates.) "Inductively Coupled Plasma – Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes, Method 200.7"
EPA CLP SOW	U.S. EPA. Contract Laboratory Program Statement of Work for Organic and Inorganic Analysis, Multi-Media, Multi-Concentration. Documents number ILM05.4, **and** SOM01.2.
EPA MCAWW March 1983	U.S. EPA. (March 1983.) "Methods for Chemical Analysis of Water and Wastes". EPA-600/4-79-020.
Standard Methods, 19th, 20th Ed	APHA, AWWA, WEF. (Standard Methods for the Examination of Water and Wastewater", 19th, 20th Editions 1995, 1998.
Lachat	Lachat Instruments. "Method Manual for the QuikChem Automated Ion Analyzer". Milwaukee, WI, (periodic updates by method).
SW-846 3rd Edition	U.S. EPA (September, 1986) "Test Methods for Evaluating Solid Waste Physical/Chemical Methods", SW846, Third Edition (9/86), Update I (7/92), Update II (9/94), Update III (12/96), Update IV (2/07).
EPA/6006R-93/100	U.S. EPA "Methods for the Determination of Inorganic Substances in Environmental Samples", Method 300.0, Revision 2.1, August 1993
EPA-821-R-98-002	"Method 1664, Revision A: n-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated n-Hexane Extractable Material (SGT-HEM; Non-polar Material) by Extraction and Gravimetry", EPA-821-R-98-002, February 1999
RSK-175	Dissolved Oxygen and Methane in Water by a GC Headspace Equilibration Technique", R.S. Kerr Environmental Research Laboratory, Ada, OK, RSK-175, March 15, 1989
EPA/4-81-054, page 115	U.S. EPA EMSL Cincinnati, Ohio, September 1978, "Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater, page 115, "Method for Chlorophenoxy Acid Pesticides in Water and Wastewaters."
Lloyd Kahn Method	"Determination of Total Organic Carbon in Sediment," July, **1988 modified for soils**

3.0 Quality Assurance Objectives

The overall QA objective for CompuChem is to develop and implement procedures for laboratory analysis, chain-of-custody, and reporting that will provide results that are of known and documented quality. Data Quality Indicators (DQIs) are used as qualitative and quantitative descriptors in interpreting the degree of acceptability or utility of data. The principal DQIs are precision, bias (accuracy), representativeness, comparability, completeness and detection limits. DQIs are used as quantitative goals for the quality of data generated in the analytical measurement process. This section summarizes how specific QA objectives are achieved. The specific application of these various activities are contained in the method SOPs.

3.1 Precision and Accuracy

Statistically derived control limits, determined from laboratory-acquired historical data, are reviewed annually and updated if needed. These limits are used for the evaluation of the laboratory control sample (LCS) in the control chart program and at the bench for some procedures. For other procedures, control limits are established in the method, Statement of Work (SOW), or laboratory Standard Operating Procedures (SOPs).

Statistical control limits for matrix spikes and matrix spike duplicates (MS/MSD) have been generated for SW-846 and wet chemistry methods. Default limits have been established internally when statistical limits are broad. For matrix spikes and for laboratory control samples the default limits are 20-150%, with allowances for marginal exceedances.

While the statistical limits presented are goals to be achieved, a specific client or project may dictate the use of other limits. In these cases the laboratory exercises flexibility in applying these in order to meet client-specific requirements. For the Contract Laboratory Program (CLP), matrix spike recoveries are specified in the SOW.

An initial precision and accuracy demonstration is performed for each method. Four replicate control samples are spiked at concentrations near the calibration midpoint and processed through the entire analytical method. The mean percent recovery and percent relative standard deviation are calculated from the replicate results. Precision and accuracy studies are used to document analyst proficiency in performing methods. Data from these studies are maintained **by** the QA department and **electronically in** training files. The standard used in the preparation of the precision and accuracy determinations generally has a concentration within 20% to 80% of the linear calibration range.

The accuracy limits for surrogates are specified by the method or determined statistically for SW-846 and are applicable to laboratory QC samples**,** as well as field samples.

3.1.1 Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Precision is assessed through the calculation of relative percent differences (RPDs) and relative standard deviations (RSDs) for replicate samples. For inorganic analyses, laboratory precision is assessed through the analysis of a sample/sample duplicate pair, a matrix spike/matrix spike duplicate (MS/MSD) pair, and/or field duplicate pairs. For organic analyses, precision is assessed through the analysis of MS/MSD and field duplicate samples.

When acceptance criteria for precision are not met, depending on the type of problem and sample holding time, corrective action may involve reporting data with a laboratory qualifying notice. An example is the poor precision between matrix spike duplicates when one of the extracts is not concentrated accurately or when matrix interference is evident. When an analyte does not meet criteria for RPD in inorganic duplicates, a data qualifying flag accompanies the result in associated samples in accordance with the U.S. EPA CLP reporting convention.

Formulas used to calculate precision of test measurements and the associated acceptance ranges are as follows.

3.1.1.1 Percent relative standard deviation (%RSD)

$$\% \text{ RSD} = \frac{s}{x} \times 100$$

where: x = average of the data points,
s = is the standard deviation

3.1.1.3 Relative Percent Difference (RPD)

$$\text{RPD} = \frac{|A-B|}{1/2\,(A+B)} \times 100$$

where: A = conc**entration** in sample A
B = conc**entration** in Sample B

3.1.2 Accuracy

Accuracy is the degree of agreement between an observed value and an accepted reference or true value.

Accuracy is assessed by the analysis of quality control samples and through the adherence to all sample handling and holding time requirements. Accuracy is demonstrated through the recovery of spiked compounds in the analysis of the MS/MSD and laboratory control samples (LCS), through surrogate recoveries in organic samples and QC **samples**, and through quality control check samples with known concentrations of target analytes.

3.1.2.1 Surrogate, Deuterated Monitoring Compounds and Spike Standard Recoveries

Evaluating the recoveries of spike, Deuterated Monitoring Compounds (DMC) and surrogate standards is a means to assess method accuracy in a specific sample matrix when subjected to specific method conditions. Samples are fortified before extraction, purging, and digestion or distillation.

The recovery of these standards is quantitatively measured during analysis. Records of the percent recovery for laboratory control samples are maintained in control chart databases. The statistical warning and control limits are updated annually, if needed. Surrogate, DMC, and spike compound recoveries must meet acceptance criteria before the

analytical data are reported. In some instances the sample matrix may produce interferences that adversely affect recoveries of surrogates, DMC or spike compounds. Re-preparation and/or reanalysis of the sample must confirm the results. When a matrix spike test fails spike recovery criteria, the LCS must be evaluated. If the LCS test fails, the entire batch must be reprocessed. If the LCS passes, the sample matrix effect is confirmed. The data **are** qualified in the narrative or with a flag.

The formulas used to calculate accuracy of test measurements follow:

3.1.2.2 Accuracy = Percent Recovery (%R)

$$\%R = \frac{SSR - SR}{SA} \times 100$$

where: SSR = conc**entration** of spiked sample
SR= conc**entration** in unspiked sample
SA = expected conc**entration** of spike

3.1.2.3 Control limits are calculated using the following formulas:

LCL = x – 3sd where: LCL = lower control limit
UCL = x + 3sd UCL = upper control limit
x = mean percent recovery
sd = standard deviation using n-1 degrees of freedom

3.2 Table of Concentration Levels for QC Samples

Type	*Purpose*	*Conc. Level*	*Method Reference*
MS/MSD	**Precision/Accuracy**	**Mid Level**	**CLP organics** **SW-846 organics** **Wet Chemistry**
MS	**Accuracy**	**Mid Level**	**Inorganics**
BS/LCS	**Accuracy**	**Mid Level**	**All analyses**
Duplicate	**Precision**	**Sample-dependent**	**Inorganics** **Wet Chemistry**

Mid level = Mean level between the minimum detection level and the upper end of the linear range

3.3 Representativeness

Representativeness expresses the degree to which the analytical results of one sample accurately and precisely represent results characteristic of a population, parameter variations at a sampling point, a process or an environmental condition within a defined spatial and/or temporal boundary. Representativeness is a qualitative characteristic, and is considered a goal to be achieved rather than a quantitative measurement.

Representativeness is dependent upon both the sampling program design and proper laboratory protocol.

For many sample types, careful collection planning, sample compositing, and/or sample splitting can accomplish true representativeness. Soil samples and samples of complex or heterogeneous matrix usually present the greatest difficulties for samplers and analysts. The sampler should make every effort to homogenize the sample during collection. Laboratory chemists must, whenever practical, homogenize or thoroughly mix the sample before removing aliquots for analysis. CompuChem's sample preparation SOPs include specific procedures for homogenizing as-received samples. Volatile soil samples may not be composited or mixed**, unless otherwise specified by the client,** due to the potential loss of analytes.

Representativeness is ensured by using the proper **field collection techniques,** analytical procedures, appropriate methods, meeting sample holding times, and analyzing field duplicate samples.

The manner in which the data are correlated to the particular sampling episode and sample site are major considerations when evaluating representativeness. When the laboratory is aware of conditions adversely affecting data representativeness, a QA Notice or Laboratory Notice is included in data packages to qualify results or discussion is made in the Sample Delivery Group (SDG) narrative to provide guidance in interpreting data usability.

3.4 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected under normal conditions.

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. CompuChem's objective for completeness is to provide unqualified data of the highest quality for 100% of samples.

Factors beyond the laboratory's control that adversely affect attainment of completeness objectives include:

- receipt of samples in broken containers
- receipt of samples whose **Chain** of **Custody** (COC) or integrity is compromised in some way
- receipt of samples with insufficient volume to perform initial analyses or repeat analyses should initial efforts not meet QC acceptance criteria
- receipt of improperly preserved samples
- receipt of samples held in the field for longer than expected so that holding times are jeopardized
- receipt of incomplete or inaccurate information resulting in the application of incorrect methods
- assessment of sample data by end-users using criteria other than those stated in applicable method references or applicable data validation guidelines (Data validation guidelines may reject data although method criteria are met.)

When requested, the completeness of an analysis can be documented by including in the test report sufficient information to allow the data user to assess the quality of the results.

This information may include such items as chromatograms, mass spectra, a summary of QC sample results, and the tabulated analytical results.

Additional results, up to and including all data sufficient to recreate the entire analytical process, are optional deliverable items. These may include laboratory worksheets, calibration data, all QC sample data, and internal COC documents. The highest level document emulates that required under the U.S. EPA CLP and is intended as a legally defensible document in itself. The raw data are archived and stored electronically by scanning using Adobe Acrobat. Scanning allows for the accurate duplication of documents including signatures, affidavits, and all other information. The electronic data **are** then indexed for tracking on a **server** that is backed up daily. The images of the data are stored on duplicate data tape cassettes. One cassette is kept with the computer system for easy retrieval of data and the duplicate cassette is kept in a secure off-site location. The original hard copy report is shipped to the client, but a hard copy can be retained if requested or required.

Archived data are stored for a minimum of five years. Additional storage time may also be requested by the client for certain projects or required under certain programs such as the Ohio Voluntary Action Program (VAP). It is the responsibility of the laboratory project manager to oversee the extended retention of data reports. The auxiliary data field in the LIMS is able to record data retention time requirements for each client and to assist in tracking extended retention times. For the Ohio VAP, the laboratory must notify the agency by certified mail when the 10-year retention period has expired and retain the data until directed in writing by the agency whether they will, or will not, retain the data.

3.5 Comparability

Comparability is an expression of the confidence with which one data set can be compared with another. The analytical results can be compared to results of other laboratories because CompuChem's objectives for comparability are to:

- Demonstrate traceability of analytical/calibration standards to NIST, EPA, or other certified sources
- Use routine and approved analytical methodology
- Achieve sample holding time
- Adhere to instrument tuning and calibration procedures and frequency requirements
- Determine detection limits using Federal Register methodology consistently
- Apply appropriate levels of QC within the context of the QA program
- Report results in common units and use consistent rules in reporting data
- Participate in interlaboratory studies and independent proficiency testing programs to document laboratory performance

By using traceable standards and standard methods, the analytical results can be compared to other laboratories operating similarly. The internal QA program documents laboratory QC performance, and the inter-laboratory studies document performance compared to other laboratories.

3.5.1 QC Reference Standards Traceability

Calibration standards are traceable to the National Institute of Standards and Technology (NIST) or the EPA. EPA standards are purchased with certificates of purity and traceability.

Standards are received as neat materials and concentrated solutions with certificates of analysis (COA) for dilution into intermediate and working standard solutions.

Neat materials are logged into a receipt log and are assigned a sequential number. That number is placed on the COA and bottle label. Intermediate and working standards prepared from these materials are entered into a standard preparation logbook using the appropriate receipt log numbers to identify the components.

Concentrated standards are identified using the names and/or lot numbers which appear on the COA. Intermediate and working standards prepared from these solutions are entered into the standard preparation logbook using the name and/or lot number to identify the components.

In the standard preparation logbook, intermediate and working standard solutions are given unique identification numbers (lot numbers). These lot numbers are used to reference these solutions on all subsequent data such as instrument logs and sample preparation sheets.

Regarding standard(s) preparation, the following statement is contained in all appropriate SOPs: "all spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration."

COA for all standards are retained for traceability to NIST sources. All standard containers (neat materials, concentrated solutions, intermediate and working standards) are labeled with a lot number, preparation/receipt date, expiration date, and analyst's initials.

3.5.2 Proficiency Tests

Interlaboratory studies in the form of proficiency tests are performed on at least a semiannual basis (NELAC) or quarterly (EPA).

3.6 Limits of Detection and Reporting Limits

Method Detection Limits (MDLs) are determined for all analytes as specified in the NELAC standards.

3.6.1 Method Detection Limits

A statistical method detection limit (MDL) study **or MDL verification study** is performed yearly for all approved methods in use. The studies are performed following the design specified in the Federal Register, 40 CFR Part 136 (October 26, 1984).

The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

The MDL is determined from the analysis of a sample in a lab pure matrix containing the analyte.

At least seven replicate samples are prepared containing the analyte(s) to be tested at a concentration that is 3 to 5 times the estimated MDL for each analyte. The samples are processed through the entire analytical method.

The MDL is calculated using the standard deviation of the replicate measurements and the Student's T value at the 99% confidence level (n-1 degrees of freedom). The mean analyte value is also calculated. The Dixon outlier test or a similar one is used to eliminate any anomalous data point, but a minimum of seven replicate values are required for calculating the MDL.

Detailed MDL criteria are presented in the **method** specific SOP.

3.6.1.1 MDL Calculation

- MDL = $t(n-1, 1-\alpha = .99)(\sigma n-1)$

  where: $t(n-1, 1-\alpha = .99)$ = the student's value for a 99% confidence level and a standard deviation with n-1 degrees of freedom

  $\sigma n-1$ = standard deviation of the replicate analysis

- Standard Deviation

$$\sigma n-1 = \sqrt{\frac{\sum (x_i - x)2}{n-1}}$$

where: x_i = the data point
x = the mean
$\sigma n-1$ = standard deviation
n = number of samples

3.6.2 Instrument Detection Limits

To fulfill the requirements of **certain methods**, instrument detection limit (IDL) determinations are performed quarterly. This is accomplished by the analysis of seven replicate standards prepared in reagent water containing all target analytes at a concentration of 3 to 5 times the estimated IDL. These analyses must be performed over three non-consecutive days and the standard deviations of the replicates calculated. The IDL is calculated by multiplying the average of these standard deviations by three.

For SW-846 ICP analysis, the calculated IDL value must be less than or equal to **the** MDL for the analyte.

3.6.3 Method Detection Limit Default Cut-off Values

For organic methods, when the concentration of a detected target analyte is between the MDL and the reporting limit, it is reported with a "J" flag to indicate the result is estimated.

For projects that do not require the results to be reported down to the MDL, a cut-off value has been defined by the laboratory and may be used. The definition states that any analyte with an MDL <1/5 the RL will have a default cut-off value equal to 1/5 the RL. Any analyte with an MDL >1/5 the RL will have its actual MDL as the cut-off value. For the latter, the MDL has to be less than the RL.

Clients would receive prior notification from the project manager for analytes falling into the latter scenario and may require an elevation in the reporting limit.

The use of the MDL cutoff option is not utilized for the EPA Contract Laboratory Program Statement of Work or clients requiring the actual MDL value.

3.6.4 Reporting Limits

For CLP methods, the reporting limit is the Contract Required Quantitation Limit (CRQL). Results are reported down to the MDL for ILM05.4 and organic parameters. Results reported between the CRQL and the MDL are flagged to indicate the quantitated results are estimated.

For non-CLP methods using a multipoint calibration, the reporting limit is equivalent to or higher than the lowest calibration standard concentration. For ICP metals, the laboratory determines a practical quantitation limit (PQL) for each analyte to be used as the reporting limit..

3.7 Limit of Detection (LOD) for DoD Projects: Determination and Verification

The method detection limit as described in Section 3.6.1 is used to determine the LOD for each analyte, including surrogates, and matrix, as well as for all preparatory and cleanup methods routinely used on samples, as follows:

After each detection limit determination, the LOD is established immediately by spiking a quality system matrix at approximately two to three times the detection limit (for a single-analyte standard) or one to four times the detection limit (for a multi-analyte standard). This spike concentration establishes the LOD. It is specific to each combination of analyte, matrix, method (including sample preparation), and instrument configuration. The LOD is verified quarterly **for each quality system matrix and annually for each quality system matrix, method, and analyte**. The following requirements apply to the initial detection limit/LOD determinations and to the quarterly LOD verifications.

- The apparent signal to noise ratio at the LOD must be at least three and the results must meet all method requirements for analyte identification (e.g., ion abundance, second-column confirmation, or pattern recognition). For data systems that do not provide a measure of noise, the signal produced by the verification sample must produce a result that is at least three standard deviations greater than the mean

method blank concentrations.

- The LOD is verified on each instrument used for a given method.
- If the LOD verification fails, then two consecutive LOD verifications at a higher concentration are performed. If they pass, the LOD is then set at the higher concentration. If they fail, the detection limit determination and LOD verification are repeated at a higher concentration and the LOD is then set at the higher concentration
- All documentation for all detection limit determinations and LOD verifications is maintained by the laboratory.
- Detailed LOD criteria are presented in the specific **method** SOP.

3.8 Limit of Quantitation (LOQ) for DoD Projects: Establishment and Verification of LOQ

For DoD projects, the LOQ is set within the calibration range prior to sample analysis. The LOQ is verified quarterly **at a minimum**.

For methods using a multipoint calibration, **the** Limit of Quantitation (LOQ) is equivalent to or higher than the lowest calibration standard concentration. For ICP metals, the laboratory determines a practical quantitation limit (PQL) for each analyte to be used as the Limit of Quantitation (LOQ).

Four replicate control samples are spiked at concentrations at the LOQ and processed through the entire analytical method. The mean percent recovery and percent relative standard deviation are calculated from the replicate results. The LOQ and associated precision and bias must meet SOP and client requirements and is reported. If the method is modified, precision and bias at the new LOQ is demonstrated and reported.

Detailed LOQ criteria are presented in the specific **method** SOP.

3.9 Method Validation Studies

Prior to analysis of any samples for approved methods not previously offered for sale by the laboratory, a method validation study must first be performed. A chemist familiar with the extraction/preparation procedures and the instrumental detection systems reviews the as-written method. The chemist looks for safety hazards, applicability of available instrument systems, new equipment requirements, any discrepancies in the written method, and the QA/QC requirements. A plan of testing approach is discussed with the laboratory director, technical directors, and executive management. **Analyses** of laboratory spiked matrices **are** performed to determine overall method recoverability. A draft SOP is written after the method validation study has been performed.

Documentation includes an MDL study, a precision and accuracy determination, and a draft SOP. This and any other pertinent information**, such as published methodologies,** is then forwarded to the QA department for final approval. Any deviations from the published method must be noted in the SOP. Once approval by QA and SOP document control **are** completed, product codes can be developed and the new method can be offered to clients.

3.10 Data Quality Objectives

Data quality objectives (DQOs) are used in planning environmental data collection activities. They establish the level of data that is needed to support decisions regarding the site. They establish the level of uncertainty in results that a decision-maker is willing to accept. They can be used to define QA/QC programs specific to a project or data collection activity. DQOs have been established for programs under which the laboratory provides analytical services. Internal project support staff work closely with the client and regulatory agencies to ensure that DQOs will be met by the analytical results provided. The project management team at CompuChem conveys project and client-specific requirements to the laboratory by using the Laboratory Information Management System (LIMS).

4.0 Sampling Procedures

CompuChem does not perform field sampling services. We provide pre-preserved glassware with guidance on collection procedures, if requested, in compliance with program-specific and regulatory shipping requirements. Preservation and holding time requirements are listed in Tables 4.1 through 4.7. We offer shipping supplies that include a shipping container and requested glassware. Glassware supplied by CompuChem for sample collection is purchased pre-cleaned and certified from the vendor. Cleaning procedures are performed according to the U.S. EPA Office of Solid Waste Emergency Response (OSWER) directive published in the document *Specifications and Guidance for Obtaining Contaminant-Free Glassware* (current approved version). The vendor also provides results of analyses using low detection limit EPA methods to certify the glassware is free of contaminants. Certificates of analysis accompanying the glassware are kept on file in the Receiving department and made available upon request.

4.1 Quality Control Procedures

4.1.1 Quality Control of Bottles

Pre-cleaned and certified glassware lots, including all of the bottles, jars, and vials sent to clients for the purpose of sample collection, are purchased and stored at CompuChem. This glassware arrives at the laboratory with certificates of analysis **for each lot and** documents its level of contamination. The level of contamination must be less than the reporting limit (< ½ the reporting limit for **DoD** projects). Preservative is added to sample collection bottles prior to shipment to the client with the exception of volatile vials. Volatile **glassware i**s received from the vendor containing preservative. Individually packaged EnCore™ sampling devices are provided for collection of 5 gram volatile soil samples for use with Method 5035 **sampling (including GRO)** and certain methanol preserved TPH analyses. **Terra Core sampling kits are also available.**

4.1.2 Quality Control of the Pure Water System and Vendor Pre-Cleaned and Certified Bottles

A U.S. Filter pure water system is used to generate ASTM Type II reagent grade water. This laboratory pure water is used in sample and method blank preparations.

In addition, we supply water to our clients for use as a source of field QC samples (trip blanks, rinsate blanks, etc.) This water is stored in the volatile instrumentation laboratory in a series of 45 liter glass carboys. Water used for this purpose and for the volatile analytical procedures is generated by a Millipore system located in the volatile sample preparation room.

For many years, the water collected in each carboy **had** been analyzed for volatile, semivolatile, pesticide/PCB and inorganic (metal and cyanide) analytes. An assessment of the results from these tests has demonstrated that, with the exception of common laboratory solvents, the water had not contained target analytes above any blank acceptance criteria. For that reason, the analytical testing of the carboy water was discontinued, unless requested by a client.

Water used to prepare laboratory QC samples, such as method blanks and LCS, must meet method **blank QC acceptance** requirements for acceptability.

4.1.3 Volatile Storage Blank Tests

Storage blank tests associated with CLP analyses and certain commercial clients analyses are performed on a routine basis. The storage blank is used to demonstrate that the refrigerated storage environments, for samples submitted for volatile organic analyses, are free from contamination. Cooler tests are not required for any refrigerators being used strictly for long-term storage of volatiles samples.

4.2 Shipping Coolers

4.2.1 Preparation

An Igloo-style container is provided as a service to customers who request that CompuChem supply bottles to the field **for sample collection**. A Customer Service representative determines, with the client, the required number and types of bottles and fills out a cooler request form (Figure 4-1) that includes the following information:

- address of the client
- special instructions: use of chain-of-custody **(COC)**, etc.
- method of shipment
- account number
- latest shipping date
- test codes for samples

The cooler is sent with instructional and necessary information such as**: COC** record (Figure **4**-1), sample collection and preservation guidelines (Figure 4-2), bottle labels, custody seals (Figure 4-3), etc. When soil samples are to be imported into the USA, the US Department of Agriculture requires the receiver hold a valid permit to receive soil (Figure 4.4) and that the sample containers be labeled with "Restricted Entry" labels (Figure 4-5). CompuChem encloses these labels and instructions when sent outside the continental U.S.

For regulatory work done in the States of Florida and New Jersey and for some other clients, formal COC starts when the pre-cleaned sample containers are dispatched to the field from the laboratory. The required COC record is sent with these containers. It must be signed and dated by the individual who packs the cleaned and certified sample containers into the cooler and relinquishes custody of them. This initiates the COC process.

Protocol for sample collection is left to the discretion of the field sampling crew. In general, the use of "blue" ice for sample preservation is discouraged and is adequate <u>only</u> if the samples have been pre-cooled with wet ice.

Please note that Tables 4.1 to 4.7 list Preservation, Holding Times, and Recommended Sample Volumes published in a variety of federal documents. CompuChem does not perform all these methods in-house but supplies this

information for the benefit of our clients. See **Section** 2 of the Quality Manual for details on analyses performed at CompuChem.

Table 4.1

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the Clean Water Act, 40 CFR 136, *Federal Register, March 12, 2007*

(For a more detailed explanation of Holding Times, refer to note at end of table footnotes[1], page 5.)

Parameter	Preservation	Holding[a] Time (days)	Containers[b]	Volume (mL)
Acidity	Cool, ≤ 6°C[g]	14	P or G	200
Alkalinity	Cool, ≤ 6°C[g]	14	P or G	100
Ammonia	Cool, ≤ 6°C[g], H_2SO_4 to pH<2	28	P or G	500
Biochemical Oxygen Demand	Cool, ≤ 6°C[g]	48 hours	P or G	1000
Bromide	NR[c]	28	P or G	200
Chemical Oxygen Demand	Cool, ≤6° C[g], H_2SO_4 to pH<2	28	P or G	100
Chloride	NR	28	P or G	100
Chlorine, Total Residual	NR	0[d]	P or G	500
Chromium VI	Cool, ≤ 6°C[g]	24 hours	P or G	500
Coliform, fecal and total	Cool, < 10°C; 0.008% $Na_2S_2O_3$	6 hours	P or G	200
Color	Cool, ≤ 6°C[g]	48 hours	P or G	500
Cyanide, total	Cool, ≤ 6°C[g]; NaOH to pH>12 0.6 g ascorbic acid[f]	14[e]	P or G	1000
Cyanide, amenable to chlorination (free)	Cool, ≤ 6°C[g]; NaOH to pH>12 0.6 g ascorbic acid[f]	14[e]	P or G	500
Fluoride	NR	28	P	500
Hardness	HNO_3 to pH<2 H_2SO_4 to pH <2	180	P or G	250
Hydrogen Ion (pH)	NR[c]	0[d]	P or G	40
Kjeldahl Nitrogen	Cool, ≤ 6°C[g]; H_2SO_4 to pH <2	28	P or G	1000
Mercury	HNO_3 to pH<2	28	P or G	500
Metals (except Cr VI and Hg)	HNO_3 to pH<2	180	P or G	500
Nitrate (as N)	Cool, ≤ 6°C[g]	48 hours	P or G	100
Nitrite (as N)	Cool, ≤ 6°C[g]	48 hours	P or G	50
Nitrate-Nitrite (as N)	Cool, ≤6°C[g]; H_2SO_4 to pH <2	28	P or G	500
Oil and Grease	Cool, ≤ 6°C[g]C; H_2SO_4 to pH <2	28	G	1000
Organic Carbon, Total (TOC)	Cool, ≤ 6°C[g]°C HCl or H_2SO_4 to pH <2	28	P or G	100
Organic Nitrogen, Total	Cool, ≤ 6°C[g]; H_2SO_4 to pH <2	28	P or G	500
Orthosphosphate (as P)	Filter immediately, Cool, ≤ 6°C[g]	48 hours	P or G	50
Phenols (Total)	Cool, ≤ 6°C[g]; H_2SO_4 to pH <2	28	G	1000

Parameter	Preservation	Holding[a] Time (days)	Containers[b]	Volume (mL)
Phosphorus (Elemental)	Cool, ≤ 6°C[g]C	48 hours	G	500
Phosphorus (Total)	Cool, ≤ 6°C[g] H_2SO_4 to pH <2	28	P or G	200
Solids (Total and Filterable)	**Cool, ≤ 6°C**	**7**	**P or G**	**100**
Solids (Non-filterable)	**Cool, ≤ 6°C**	**7**	**P or G**	**300**
Solids (Settleable)	**Cool, ≤ 6°C**	**7 days**	**P or G**	**1000**
Silica	**Cool, ≤ 6°C**	**28**	**P**	**100**
Specific Conductance	**Cool, ≤ 6°C**	**28**	**P or G**	**250**
Sulfate	**Cool, ≤ 6°C**	**28**	**P or G**	**250**
Sulfide	**Cool, ≤ 6°C** **Add zinc acetate and NaOH to pH>9.**	**7**	**P or G**	
Sulfite	**NR[c]**	**0[d]**	**P or G**	**250**
Surfactants	**Cool, ≤ 6°C**	**48 hours**	**P or G**	**250**
Turbidity	**Cool, ≤ 6°C**	**48 hours**	**P or G**	**250**
Purgeable Halocarbons	**Cool, ≤ 6°C** **0.008% $Na_2S_2O_3$[e]**	**14**	**G**	**80**
Purgeable Aromatic Hydrocarbons	**Cool, ≤ 6°C** **0.008% $Na_2S_2O_3$[e]** **HCl to pH<2**	**14**	**G**	**80**
Phenols	**Cool, ≤6°C** **0.008% $Na_2S_2O_3$[e]**	**7/40[f]**	**P or G**	**2000**
Benzidines	**Cool, ≤ 6°C** **0.008% $Na_2S_2O_3$[e]**	**7/7[g]**	**P or G**	**2000**
Phthalate Esters	**Cool, ≤ 6°C**	**7/40[f]**	**G**	**2000**
Nitrosamines	**Cool, ≤ 6°C, dark** **0.008% $Na_2S_2O_3$[e]**	**7/40[f]**	**P or G**	**2000**
PCBs	**Cool, ≤ 6°C**	**7/40[f]**	**P or G**	**2000**
Nitroaromatics and isophorone	**Cool, ≤ 6°C, dark** **0.008% $Na_2S_2O_3$[e]**	**7/40[f]**	**P or G**	**2000**
Polynuclear Aromatic Hydrocarbons	**Cool, ≤ 6°C, dark** **0.008% $Na_2S_2O_3$[e]**	**7/40[f]**	**G**	**2000**
Haloethers	**Cool, ≤ 6°C** **0.008% $Na_2S_2O_3$[e]**	**7/40[f]**	**P or G**	**2000**
Chlorinated Hydrocarbons	**Cool, ≤ 6°C**	**7/40[f]**	**P or G**	**2000**
TCDD	**Cool, ≤ 6°C** **0.008% $Na_2S_2O_3$[e]**	**7/40[f]**	**P or G**	**2000**
Pesticides	**Cool, ≤ 6°C; pH 5-9**	**7/40[f]**	**P or G**	**2000**

[a] From time of sample collection
[b] Polyethylene (P) or glass (G)
[c] None required
[d] 0 days indicates that the sample must be analyzed immediately.
[e] Reduced to 24 hours if sulfide is present, unless sulfide is removed before preservation.
[f] Added if residual chlorine is present
[g] Aqueous samples must be preserved at ≤6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulating authority. Also, for purposes of NPDES monitoring, the specification of "≤°C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods.

[1] **Please note: all holding times without an "f" footnote are holding times from sample collection time until time of analysis. For "f" or "g" footnoted analytes, the number 7 refers to days from collection to extraction. The number following the 7 is the holding time in days from extraction to analysis.**

.Table 4.2

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the U.S. EPA CLP SOW for Inorganics Analysis ILM05.4

Parameter	Preservation[a]	Holding Time[b] (days)	Containers[c]	Volume (mL)
Cyanide, total	Cool, 4°C NaOH to pH>12 0.6 g of ascorbic acid[d]	12	P or G	1000
Metals (except Hg)	HNO_3 to pH<2, 4°± 2°C	180	P or G	500[e]
Mercury	HNO_3 to pH<2, 4°± 2°C	26	P or G	500[e]

[a] Water sample only; preservation performed by sampler immediately upon sample collection. Soil/sediment samples are maintained at 4 °C until analysis. Dissolved metals are filtered onsite by sampler before addition of preservative. Water samples received under the ILM04.1 SOW are stored at ambient temperature
[b] From validated time of sample receipt
[c] Polyethylene (P) or glass (G)
[d] Only used in the presence of residual chlorine
[e] Can be combined into a one-liter bottle.

Table 4.3

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the U.S. EPA CLP SOW for Organics Analysis SOM01.2

Parameter	Preservation	Holding Time (days)[a]	Containers[b]	Volume (mL)
Aqueous Volatiles	Cool, 4°± 2°C[e] HCl to pH ≤2	10[d]	G-TLSSL	80
Soil/Sediment Volatiles	4°± 2°C[c], sodium bisulfate < -7°C	10[d] 10	G-TLC, CET, EnCore™ samplers	4 oz 60 mL
Aqueous Semivolatiles	4°± 2°C[c]	5/40[e]	AG	2000
Soil/Sediment Semivolatiles	4°± 2°C[c]	10/40[e]	G	8 oz
Aqueous Pesticides/PCBs	4°± 2°C[c]	5/40[e]	AG	2000
Soil/Sediment Pesticides/PCBs	4°± 2°C[c]	10/40[e]	G	8 oz

[a] From validated time of sample receipt
[b] All containers are 1-liter amber glass (AG) bottles with Teflon-lined cap except aqueous volatiles (G-TLSSL = 40-ml glass bottle with Teflon-lined septum sealed lid), and soil/sediment volatiles (G-TLC = 4-oz glass jar with Teflon-lined cap or CET = closed-end tubes such as brass sleeves). Soil samples may also be collected under SOM01.2 for low level analysis in Encore™ samplers or as 5 gm sample in sodium bisulfate or medium level analysis in pre-weighed vials containing 10ml methanol. When this occurs, the sample vial, with 10 ml methanol and all labeling, is weighed to the nearest 0.1g prior to the addition of sample. Approximately 5 g of sample is added to the vial. The sample vial with sample is weighed to the nearest 0.1 g. The initial weight, final weight and sample weight are recorded and provided to the laboratory.
[c] Preserve samples at time of collection; samples should be stored in the dark until extraction/analysis.
[d] Until analysis
[e] To complete extraction/to complete analysis following extraction

Table 4.4

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Aqueous and Solid Matrices[a] Published in SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Update IV, February 2007.

Parameter	Methods	References	Container	Preservation	Maximum Holding Times[b]
Volatile Organics in water	Purge and trap GC and GC/MS	8015A modified/ 8015B, 8015C 8260B	Glass, 40-ml vial with zero headspace	Cool to ≤ 6°C, HCl to pH <2, 0.008% $Na_2S_2O_3$	14 days (A) (see below for soil)
Semivolatile Organics	GC, HPLC, and GC/MS	8081A, 8081B, 8082, 8082A, 8151A, 8310, 8270C, 8270D, 8330, 8330A, 8332	Glass, amber (A) (1 liter sample); 8-oz wide mouth with PTFE-lined cap (S) (50-g min. ea. test)	Cool to ≤ 6°C, 0.008% $Na_2S_2O_3$	7 days to extract (A); 14 days to extract (S); 40 days to analyze (A/S)
Total Metals – except Mercury and Chromium VI	ICP AES, ICP/MS	6010B, 6010C, 6020, 6020A	Glass or plastic 500ml (A), 8-oz wide mouth (S) (200-g sample)	HNO_3 to pH<2 (A); filter dissolved on-site first; filter suspended-no acid	6 months (A/S)
Chromium VI	Colorimetric, automated	7196A	Glass or plastic, 500ml (A), 8-oz wide mouth (S) (200-g sample)	Cool to ≤ 6°C	24 hours (A); one mo. to extraction, 4 days after (S).
Mercury	Manual cold vapor AA	7470 7471A 7471B	Glass or plastic, 500ml (A), 8-oz wide mouth (200-g sample)	pH<2 HNO_3 (A); Cool to 4°C (S)	28 days (A/S)
Cyanide	Colorimetric automated	9012A	Glass or plastic	Cool to ≤ 6°C; pH >12 NaOH (A)	14days (A)
Phenols	Colorimetric, automated	420.2	Glass or plastic	Cool to ≤ 6°C; pH < 4 H2SO4 (A)	28 days (A)
Specific conductance	conductivity	9050A	Glass or plastic	NA	28 days (A)
Sulfide	distillation, titration	9034	Glass or plastic	Cool to ≤ 6°C; Zinc acetate (A/S)	7 days (A/S)
Oil & grease	Gravimetric	9070 9071A	Glass	Cool to 4°C (A/S); 5 ml diluted HCl(A)	28 days (A) 14 days (S)
TOC	Combustion analyzer	9060	Glass or plastic	Cool to ≤ 6°C; pH<2 HCl or H_2SO_4; Store in dark (A)	28 days

[a] Table originally excerpted, in part, from Table II, 40 CFR 136 Vol. 72, No. 47/Monday, March 12, 2007/Rules and Regulations.
[b] Holding time begins at time of sample collection.
[c] $Na_2S_2O_3$ added if residual chlorine is present; approx. equal to 1 ml of 10% thiosulfate / liter of sample.
(A) aqueous, (S) solid

Table 4.5

Sample Handling, Preservation and Holding Time Table for SW-846 Method 5035, Volatile Organics in Soil

Conc. Level	Sampling Device	Collection Procedure	Container Type	Container Preparation	Preservation	Maximum Holding Time[a]
≤ 200 ug/kg	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1	Cool, ≤ 6°C, $NaHSO_4$	14 days
	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1[b]	Cool, ≤ 6°C	48 hours
	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1	Cool, ≤ 6°C/-10°C[d]	48 hours/ 14 days[e]
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[b,f,g]	Cool, ≤ 6°C	48 hours
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[f,g]	$NaHSO_4$, Cool, ≤ 6°C	48 hours, 14 days
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1 [b,f,g]	Cool, ≤ 6°C /-10°C [c,d]	48 hours/ 14 days
> 200 ug/kg[h]	Encore™ or equivalent	5035-Section 6.2.2.3[f]	Encore™ or equivalent	5035-6.1.1 [f,g]	Cool, ≤6°C	48 hours 14 days
	Coring Device	5035-Section 6.2.2.3[I]	Glass vial w/ PTFE-silicon septum	5035-6.1.1 [I]	Methanol/PEG Cool, ≤ 6°C	14 days
	Conventional Devices	FLDEP SOP –Section 4.3	Glass vial w/ PTFE-silicon septum	5035-6.1.1	Cool, ≤ 6°C	14 days
Dry weight	Conventional Devices		Glass w/ teflon liner		Cool, ≤ 6°C	

[a] Maximum time allowed from time/date of collection to sample analysis.
[b] Eliminate 6.1.1.2; use only organic-free water.
[c] Contents of sampling device must be transported to the laboratory at ≤6 °C and stored at –10 °C; this option upon client request.
[d] In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle.
[e] Maximum allowed time at ≤6 °C is 48 hours; maximum allowed time to sample analysis is 14 days from collection.
[f] Conducted in the laboratory.
[g] Entire contents of sampling device is extruded into the sample analysis vial containing the appropriate solvent.
[h] Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200 µg/kg.
[i] Methanolic preservation in the field is not recommended.

Table 4.6

Requirements for Holding Times for Hazardous Characteristics Indicators Published in SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Update 3, December 1996.

Parameter	Methods	References	Container	Preservation	Maximum Holding Times
Reactivity-Total releasable cyanide	Reflux distillation, colorimetry	Chapter 7, 7.3.3, 9014	Glass, with zero headspace	≤6 °C in dark, pH>12 NaOH	ASAP
Reactivity-Total releasable Sulfide	Acid distillation, titration	Chapter 7, 7.3.4, 9034	Glass, with zero headspace	≤6°C in dark, pH>9 NaOH; zinc acetate	ASAP
Ignitability	Flash point	1010	Glass or plastic	NA	ASAP
Corrosivity	Electrometric	9040B	Glass or plastic	NA	ASAP
Paint filter liquids	filtration	9095A	Glass or plastic	NA	ASAP

Table 4.7

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Toxicity Characteristics Leaching Procedure, TCLP, Method 1311, and Synthetic Precipitation Leaching Procedure, SPLP, Method 1312, Published in SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996 and Update IV, February 2007.

Sample Maximum Holding Times				
Parameter	**From: Field collection** **To: TCLP Extraction**	**From: TCLP Extraction** **To: Preparative Extraction**	**From: Preparative extraction** **To: Determinative Analysis**	**Total elapsed time in days**
Volatiles	14	NA	14	28
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, no Hg	180	NA	180	360

Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

Holding time associated with the ASTM leachate generation method (D3987-85) is 14 days from collection to leachate generation, i.e., filtration, then the method holding time to preparation and/or analysis.

Figure 4-1

GLASSWARE/COOLER ORDER FORM

DATE: _____

REQUESTED BY: _____

CLIENT: _____

ADDRESS: _____

_____

_____

_____

PHONE #: _____

DATE NEEDED BY CLIENT: _____ (circle one) AM PM

**SHIP VIA: _____ (circle one) NDA NDAS 2nd DAY AM 2nd DAY AIR 3 DAY GROUND

PROJECT ID: _____

COOLER #s _____

BOTTLES PER ANALYSIS

Number of Samples*	Parameters Needed	Container	Bottle Lot Numbers	Pres.	Preservative Lot Number	Total Bottles

* includes QC

Circle items needed: Coc's Labels Seals ____TB's / carboy#____ ____Liters DIH2O / carboy#____

Additional items: _____

Prepared by/date: _____

** NDA = NEXT DAY AIR (by 10:30AM); NDAS = NEXT DAY AIR SAVER (by 3:00PM); 2nd DAY AM = 2nd DAY AIR AM (by 10:30AM); 2nd DAY AIR = 2nd DAY AIR (by 3:00PM); 3 DAY = 3 DAY SELECT (by 3rd DAY PM); GROUND = GROUND 3-5 DAYS (SLOW BOAT)

Figure 4-2

Sample Collection and Preservation Guidelines

Preservatives must be added in the field at the time of sample collection unless glassware are already pre-preserved. Preservatives should be recorded on the Chain-of-Custody form in the "Remarks" column on a per sample basis. For certain methods and parameters, the laboratory verifies upon receipt that the sample pH falls within an acceptable range. Improperly preserved samples or samples with pH values outside of the specified range are noted in the sample receiving documentation. The client is contacted and given the option of resampling, directing the laboratory to preserve the sample in-house, or processing the sample as it was received. To document the action taken, a Quality Assurance Notice is included in the data report for any occurrence.

When obtaining aqueous samples for the determination of volatile organics, the collector should ensure the absence of headspace by filling the 40 **mL** bottle to the top. This procedure should produce a positive meniscus across the surface of the vial. The Teflon®-lined septum should be placed gently over the sample surface, with the Teflon® side down, and the top screwed firmly on over the septum. A proper seal should be verified by inverting the sealed bottle and gently tapping on the sides with your finger, ensuring that no air bubbles appear.

The collector should designate which samples are to be used for Quality Control. For these designated samples, the recommended sample weight or volume should be doubled. If sufficient sample is not available to the laboratory, QC requirements may not be achievable. Excess sample will unnecessarily increase shipping costs.

Guidance on the EPA SW8-46 Method 5035 for the Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples.

CompuChem has the capabilities and supplies to provide any of the Method 5035 options. Because of some disadvantages, we favor the use of disposable En Core™ samplers, with zero headspace design, for the collection of soil samples for volatile analysis. The En Core™ sampler is available in 5 g and 25 g sizes. Some regulatory agencies may mandate the 5 g **sample** size. Three 5 g En Core™ samplers, with a 2 ounce jar for percent moisture, would be required per sampling site to include:

- a 5 g aliquot for the sample analysis
- a 5 g aliquot as a back-up for the sample analysis
- a 5 g aliquot for a methanol extraction used for screening and/or a high concentration analysis
- a 2 oz. aliquot for a % moisture determination

In most cases, the methanol aliquot (containing surrogates) will be used for screening purposes. Based on those results, other tests will be performed. Once aliquots of the soil sample, taken from the En Core™ sampler, are added to vials containing preservative, the remainder of the 14 day holding time is available.

During the initial transfer of sample aliquots to the vials containing the sodium bisulfate preservative solution, the presence of carbonates will be identified due to the observed effervescence. If this occurs, a 5 g aliquot of sample will be transferred to a vial containing only water. The sample will be immediately sealed and analyzed as soon as possible but no later than 48 hours from preparation. Alternatively, the analysis will be conducted on the methanol-preserved aliquot. For effervescence, with the client's approval, another option is to freeze the contents of the En Core™. Freezing the contents of En **Core™** samplers in closed system purge and trap vials with or without the addition of water is also

allowed by some EPA Regions and other regulatory agencies. The laboratory project manager conveys this type of information directly to the laboratory upon sample receipt. Advantages of use of the En Core™ include:

- No need to take or use a balance in the field
- No exposure of field personnel to corrosive sodium bisulfate or flammable/toxic methanol
- No additional shipping paperwork or cooler labeling
- No need to use the costly closed system purge and trap vials in the field
- No chance of volatile sample container breakage during shipment
- Significant decrease in man-hour requirements to complete the field sampling efforts

An alternate method 5035 option is available for collection of volatile soils uses a Terra **Core™** kit with pre-preserved vials, a disposable coring device (**Terra Core™**) and a 2 ounce jar for percent moisture. CompuChem provides 2 sodium bisulfate and one methanol pre-preserved vials, a 2 ounce jar and the disposable **Terra Core™** to the client for the collection a volatile soil sample. The advantages of this option are that the cost is lower for the client per sample and the client does not have to ship each day of sampling if the pre-preserved vials and jars are kept at 4 degrees Celsius (± 2 degrees).

Figure 4-3

Custody Seal

CompuChem
A Division Of
Liberty Analytical Corp.

CUSTODY SEAL

Signature

Date

Figure 4-4

Animal and Plant Health Inspection Service

Plant Protection & Quarantine

United States Department of Agriculture
Animal and Plant Health Inspection Service
4700 River Road
Riverdale, MD 20737

Permit to Receive Soil
Regulated by 7 CFR 330

This permit was generated electronically via the ePermits system.

PERMITTEE NAME:	Robert E. Meierer	**PERMIT NUMBER:**	P330-09-00066
COMPANY:	CompuChem	**APPLICATION NUMBER:**	P525-090127-007
RECEIVING ADDRESS:	501 Madison Avenue Cary, NC 27513	**DATE ISSUED:**	03/27/2009
MAILING ADDRESS:	501 Madison Avenue Cary, NC 27513		
PHONE:	(919) 379-4000		
FAX:	(919) 378-4050	**EXPIRES:**	**03/27/2012**

PORTS OF ARRIVAL/PLANT INSPECTION STATIONS: FL, Miami (Physical); GA, Atlanta; Various Ports of Entry Staffed by CBP-Agriculture Inspection
HAND CARRY: No

Under the conditions specified, this permit authorizes the following:
Quantity of Soil per Shipment and Treatment
Over 3 lbs

PERMIT CONDITIONS

1. This permit is issued only for Robert E. Meierer, the named permit holder with CompuChem located at 501 Madison Ave, Cary, NC 27513. This permit cannot be transferred or assigned nor does it fulfill or modify the requirements of any other federal or state regulatory authority (such as the U.S. Environmental Protection Agency, the U.S. Fish and Wildlife Service, the U.S. Food and Drug Administration, the Centers for Disease Control and Prevention, or your state's Department of Agriculture).

2. The permit holder verifies United States residency by initialing and accepting these permit conditions. If you are not a United States resident, it is unlawful for you to initial or accept these permit conditions because a USDA 525 Soil Permit can only be issued to United States residents.

3. The permit holder is solely responsible for ensuring compliance with all statutory requirements and specifically listed permit conditions. Failure to comply with the terms and conditions of this permit is cause for the following: (a) cancellation of this permit, (b) cancellation of other permits issued to the permit holder, (c) seizure and/or destruction of regulated soil, (d) denial of future permit applications by this permit holder, (e) liability for civil penalties, and (f) criminal prosecution under provisions in the Plant Protection Act.

Permit Number P330-09-00066

THIS PERMIT HAS BEEN APPROVED ELECTRONICALLY BY THE FOLLOWING PPQ HEADQUARTER OFFICIAL VIA EPERMITS.	DATE
Monica J O'Neal Monica Jones O Neil	03/27/2009

WARNING: Any alteration, forgery or unauthorized use of this Federal Form is subject to civil penalties of up to $250,000 (7 U.S.C.s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C.s 1001)

Page 1 of 3

Figure 4-4 (Continued)

Animal and Plant Health Inspection Service

Plant Protection & Quarantine

4. Any alteration, forgery, unauthorized use of this permit and/or associated Federal Forms are subject to civil and criminal penalties including fines and imprisonment.

5. The permit holder must maintain a valid permit so long as the soil is in your possession. The permit holder must safeguard and dispose of the soil during the term of this permit. This permit cannot be extended. A new permit is required for uninterrupted authorization/use of regulated soil after this permit expires.

6. The permit holder must maintain an official permanent work assignment at the address identified on this permit. If the permit holder ceases assignment/affiliation at the address identified on this permit, or personnel circumstances change in any way, then a compliance officer must be notified at the PPQ permit unit immediately (that is, within one business day) by either (a) email to pest.permits@aphis.usda.gov, (b) fax to 301-734-4300 or 8700, or (c) conventional mail to USDA PPQ Permit Unit, 4700 River Road, Riverdale, MD 20737. The permit holder must destroy all regulated soil prior to departure unless the permit holder either (a) requests cancellation of this permit and complies with all permit-specific termination conditions, (b) applies for and receives a permit to move the soil to a new facility, or (c) relinquishes control of the regulated soil to a qualified individual who obtained a permit for the continued use of these regulated soil prior to this permit holder's departure. {rsj 04/08}

7. Without prior notice and during reasonable hours, authorized PPQ and/or State regulatory officials shall be allowed to inspect the conditions associated with the regulated article authorized under this permit.

8. This permit authorizes the importation of soil under the conditions specified below. Upon arrival into the United States, the articles, shipping container(s) and paperwork are subject to inspection by officials from Customs and Border Protection, Agriculture Inspection (CBP-AI) and USDA, APHIS, Plant Protection and Quarantine (PPQ).

9. Any person who unloads, lands or otherwise brings or moves into or through the United States any regulated plants, plant products, plant pests, soil or other products or articles in violation of the regulations will be subject to prosecution under applicable provisions of the law.

10. All foreign cargo of agricultural interest is inspected at the first port of arrival or the first port of unlading. If a shipment arrives at a port that lacks the official/personnel necessary to do the proper inspection and/or treatment, the shipment must be transferred by a bonded carrier to a nearest station staffed with proper CBP-AI or PPQ Officials at the expense of the permit holder. All subsequent movement, transfers and/or transloading, must be approved by CBP-AI.

11. A copy of this permit must accompany all shipments authorized under this permit.

12. The soil must be shipped in sturdy, leak-proof containers.

13. CBP-AI and PPQ have the authority to order and approve treatment, re-exportation or destruction of a shipment, a portion of a shipment or any other material associated with the shipment (i.e. pallets, packaging, means of conveyance). If an official of CBP-AI or PPQ determines that the shipment requires treatment as a condition of entry, is contaminated with a quarantine plant pest or pests, is commingled with prohibited plant material or the required documentation is incomplete or missing, then that official may order and approve treatment, re-exportation or destruction of a shipment, a portion of a shipment or any other material associated with the shipment (i.e. pallets, packaging, means of conveyance).

14. The shipment must be free from foreign matter or debris, plants, noxious weed seeds and living organisms such as parasitic plants, pathogens, insects, snails and mites. Material found to be commingled with unauthorized material will be subject to the same action (i.e. re-export, destruction) as unauthorized material.

Permit Number P330-09-00066

THIS PERMIT HAS BEEN APPROVED ELECTRONICALLY BY THE FOLLOWING PPQ HEADQUARTER OFFICIAL VIA EPERMITS.	DATE
Monica J O'Neal Monica Jones O Neil	03/27/2009

WARNING: Any alteration, forgery or unauthorized use of this Federal Form is subject to civil penalties of up to $250,000 (7 U.S.C.s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C.s 1001)

Page 2 of 3

Figure 4-4 (Continued)

Animal and Plant Health Inspection Service

Plant Protection & Quarantine

15. All solid wood packing material (SWPM) accompanying the shipment must be in compliance with ISPM 15 treatment regulations and IPPC stamp requirements and enforcement. Noncompliant shipments will be treated, re-exported or destroyed at the consignee's expense.

16. All costs and arrangements for safeguarding and transportation of the cargo are the responsibility of the importer, broker or other parties associated with the shipment.

17. The shipment can be released without treatment at the port of entry to the permittee's address listed on the permit or label or to an authorized user only if the final destination is an approved facility listed at https://web01.aphis.usda.gov/PPQ/AuthSoilLabs.nsf/web?openform.

18. Further distribution of soil is not allowed without prior approval from the State and Federal Regulatory Officials. Soil is to be used strictly for analysis within a laboratory at CompuChem located in Cary, NC.

19. Upon receipt, all soil samples will remain within the approved soil laboratory identified on this permit. Laboratory access is restricted to individuals authorized by the permit holder.

20. This permit does not authorize the use of soil for growing purposes and for isolation or culture of organisms.

21. All unconsumed soil, containers and effluent is to be autoclaved, incinerated or properly sterilized by the permittee at the conclusion of the project as approved and prescribed by PPQ in the compliance agreement.

22. This permit is valid for shipments of soil not heat treated at the port of entry only if a Compliance Agreement (PPQ Form 519) has been completed and signed.

23. This permit authorizes importation of soil into the United Stated from [designated foreign sources] through U.S. ports of entry staffed by a United Stated Department of Homeland Security Customs and Border Protection Agricultural Specialist.

24. You must attach a PPQ Form 550 Black/White label to the exterior of each shipment being imported under this permit. We will send you the PPQ Form 550 Black/White labels upon issuance of this permit (i.e., a signed PPQ permit) by bonded courier.
You may request additional labels by email at:
Redandwhitelabelrequest@aphis.usda.gov. You must specify the type of label you are requesting, PPQ Form 550 Black/White labels, and the number of labels needed.

You must enclose the following supplemental information with each shipment:
- Permittee Name
- Permit number

END OF PERMIT CONDITIONS

Permit Number P330-09-00066

THIS PERMIT HAS BEEN APPROVED ELECTRONICALLY BY THE FOLLOWING PPQ HEADQUARTER OFFICIAL VIA EPERMITS.	DATE
Monica J O'Neil Monica Jones O Neil	03/27/2009

WARNING: Any alteration, forgery or unauthorized use of this Federal Form is subject to civil penalties of up to $250,000 (7 U.S.C.s 7734(b)) or punishable by a fine of not more than $10,000, or imprisonment of not more than 5 years, or both (18 U.S.C.s 1001)

Page 3 of 3

Figure 4-5

Soil Sample Cooler Restricted Entry Label

CompuChem
a division of Liberty Analytical Corporation

In order to remain in compliance with current United States Department of Agriculture regulations regarding the importation of soil samples of foreign origin into the Unites States for subsequent analysis, it is necessary to affix the attached USDA labels to every soil sample that is expected to enter the U.S.A. Each label should be placed in a highly visible position on the exterior surface of every cooler being sent. Failure to comply with this regulation can result in transportation delays and denials.

U.S. DEPARTMENT OF AGRICULTURE
ANIMAL AND PLANT HEALTH INSPECTION SERVICE
PLANT PROTECTION AND QUARANTINE
4700 RIVER RD., UNIT 136
RIVERDALE, MD 20737-1236

SOIL SAMPLES
RESTRICTED ENTRY

The material contained in this package is imported under authority of the Federal Plant Pest Act of May 23, 1957.

For release without treatment if addressee is currently listed as approved by Plant Protection and Quarantine.

PPQ FORM 550
(MAR 95)

☆U.S.GPO: 1995–621-030

5.0 Sample Handling

This section summarizes policies and practices for sample **receipt** handling. Further details are contained in **receiving and sample management** SOPs. If any discrepancy is noted at sample receipt, or if there is any question about the sample's suitability for testing, the Project Manager or Customer Service Representative contacts the client for instructions on how to proceed. The condition of the samples is recorded on the Chain of Custody (COC) record, on the sample log-in form, and in the final report.

5.1 Sample Acceptance Policy

CompuChem's sample acceptance policy outlines the circumstances under which samples will be accepted by the laboratory. **Information and d**ata that do not meet the policy are noted in the laboratory report defining the nature and substance of the variation. Samples received by the laboratory for analysis require the following:

- Proper, full, and complete documentation, including:
  - unique sample identification,
  - location, date and time of collection,
  - collector's name,
  - preservation type,
  - matrix
  - analytical method
    - program area – CWA/RCRA/EPA
  - sample type- grab or composite
  - temperature of samples at time of collection for grab and at time of final aliquot collection for composite samples if required (CWA)
- special remarks concerning the sample – including references to permit specific concentration limits, if applicable
- Sample identified using durable label completed in indelible ink
- Proper preservation
  - time of preservation if required (CWA requires preservation within 15 minutes of time of collection)
- Intact, undamaged sample container
- Adequate sample volume
- Appropriate receipt temperature
- Receipt within holding time

It is the laboratory's policy to accept all samples including those that do not meet the requirements listed above. However, in cases where these requirements are not met, the project manager contacts the SMO (who contacts the EPA Region) or **other** clients via telephone or e-mail indicating any receiving issues. The laboratory will proceed with analysis or reject the sample as advised by the client or EPA Region. The laboratory cannot attest the data produced is "certified data" for Ohio VAP unless all samples are received in accordance with Ohio VAP approved document requirements.

5.2 Sample Tracking

CompuChem uniquely identifies each sample to be tested, to ensure that there can be no confusion regarding identity. The sample identification system includes identification for all samples, sub-samples and subsequent extracts and/or digestates. A unique

identification (ID) code is automatically **generated** by the LIMS **computer program**. A **unique** label is printed and affixed to the sample container.

Internal COC records accompany raw samples from the **storage** cooler to the **appropriate** laboratories and back, extracts and digestates to the instrument labs from preparation. **These records** are part of the data deliverables depending on the report style requested by the client.

5.2.1 Bench Log-In Procedure

The following steps are completed by the Receiving Clerk and Sample Custodian for all samples when received by CompuChem. If for any reason a sample requires special handling, the Project Manager is consulted for instructions for processing and documentation. Completed COC forms are part of the data deliverables.

Each employee is required to wear protective clothing (lab coat, safety glasses, and gloves) at all times when working in the **receiving** hood with samples. Upon receipt, the condition of the sample, including any abnormalities or departures from standard condition is observed and recorded. Each **shipping** container is opened carefully under the **safety** hood and inspected for damage and integrity. For clients using padlocks, sealing tape, or custody seals, these items are inspected to make sure that **the security measures** are intact and this observation is recorded on the COC form (Figure 5-1). If the custody seals, tapes, or padlocks are broken, a project manager contacts the client for instructions as to how to proceed.

The condition of the refrigerant (whether any ice remains or whether the cooling packs, if present, are solid) is checked. The temperature of a representative sample, taken from the middle of the cooler, or of the temperature blank, when present, is obtained by means of an infrared (IR) gun. The temperature is recorded on the COC. The temperature is also recorded on the log-in-sheet for EPA samples. All samples requiring thermal preservation are considered acceptable if the arrival temperature is either within +/- 2 degrees C of the required temperature (4 degrees C) or the method-specified range.

For EPA samples, the SMO has to be contacted if the as-received temperature exceeds 10 °C. Samples that are hand delivered to the laboratory immediately after collection may not meet criteria. In these cases, the samples will be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice. A Quality Assurance notice is completed and clients contacted for samples that exceed temperature requirements.

Samples are checked for breakage and removed from the shipping container. The sample identification information on the sample bottles is compared to the sample information on the traffic report, packing slip, and COC form included in the container. If discrepancies exist, the problem is noted on the COC form; the receiving clerk notifies the Project Manager.

Each 40 mL vial for volatile analysis is checked for air bubbles and headspace. Findings are noted on the Chain-of-Custody. Findings are also noted on the log-in-sheet for EPA samples. A**n air** bubble up to the size of a pea is permitted.

Incoming EPA samples are checked against SMO scheduled receipts. Samples are logged in on the log-in-sheet using either an organic or inorganic EPA Receiving Log sheet (Figures 5-2 and 5-3) for EPA samples and the following items are noted:

- Case number/SDG
- CompuChem ID number (CC#)
- Client name or order number
- temperature
- field ID (Sample ID)
- receiving date (RD)
- sampling date (SD)
- test codes
- matrix
- volume received
- pH of inorganic samples
- residual chlorine and sulfide check for cyanides

Preservation checks include a residual chlorine check performed for cyanide and phenol samples **along with a** sulfide check**.** The pH is verified for inorganic samples **by placing** a small **sample** aliquot **into a disposable sample cup and by using** a disposable pipette **to drip a small amount of the sample onto a pH test strip**. If a sample has not been properly preserved, the client is notified and a standard QA notice (Figures 5-4 through 5-12) is completed and placed in the sample file. The results are documented on the log-in sheet and on the COC.

On each complete EPA COC, Traffic Report, and commercial client COC a statement as to the condition of samples upon receipt is documented. "Good Condition" indicates that the sample cooler was received with the custody seal intact and the sample or samples were received intact with all associated documentation. If there are any discrepancies in the documentation or other problems, the exceptions are noted on the appropriate documents, initialed and dated.

The statement `Received in Good Condition' does not include sample temperature since EPA samples may be received at temperatures above the recommended 4 °C. The temperature is noted on the sample log-in sheet and COC. For EPA samples, each log-in sheet and COC is reviewed by the Receiving department supervisor or designee, who ensures that all information is properly documented. Each is signed as having been reviewed, initialed, and dated.

All COC and log-in sheets are forwarded to the Project Manager for the completion of the LIMS log-in procedures. Samples that must be preserved by refrigeration do not remain un-refrigerated for more than two hours. If the completion of order entry is delayed, the samples are placed in the walk-in cooler until labels are generated and the process can be completed.

5.2.2 Computer Log-in Procedure

For EPA and commercial samples, the Project Manage**ment Staff** enters the account data into the client setup section of the LIMS to generate the client's profile. This profile contains a listing of all requested **sample** analysis codes and project specific information.

The sample information is entered into the sample receipt portion of the LIMS in order to generate a unique laboratory identification code that consists of the work order number and a numerical suffix. EPA case and SDG numbers are associated with a specific work order number. The LIMS generated identification code (sample number) is added to the log-in sheet and the original COC form next to the client ID if possible. QC samples associated with an SDG are also assigned a unique laboratory identifier.

Sample labels print in numerical sequence based on the number of bottles received for each sample ID. The labels contain the LIMS generated identification code (sample number), client ID, receipt date, container ID, container type, and preservation. Samples are labeled by wrapping each sample bottle with its unique computer-generated label (Figure 5-13), leaving the field label exposed if possible.

The labeled samples are transferred to the appropriate **storage area**. Samples to be analyzed for extractable organics and soil inorganics are stored in cold storage units separate from samples to be analyzed for purgeable organics. For ILM05.4, aqueous inorganic samples need to be refrigerated. Standards are always stored in separate refrigerated storage units in the analytical laboratories. Aqueous inorganic samples, except for hexavalent chromium and ILM05.4 aqueous inorganics, are stored separately at room temperature.

File envelopes are used to assemble field and QC sample information for report preparation. EPA envelopes contain all information for the case including: the copy of the Organic Traffic Report (OTR), a copy of the COC, an original air bill, a copy of the log-in sheet, a copy of the EPA scheduling log, sample custody tags (if received), telephone logs, and "pull sheets" (internal chain of custody forms.) The white copy of the OTR is returned with the cover sheet to the EPA SMO. Commercial sample file folders contain the customer sample order information sheet and a copy of the COC record.

CompuChem sometimes receives raw samples from the EPA that should not have been sent to this location. These samples require a transfer. When this occurs, **and after notifying SMO,** a new COC form is filled out using the information on the sample tags. Custody is relinquished to the courier by signing and dating the "Relinquished by" section of the COC form. A copy of the COC is kept and the original COC and paperwork are sent with samples. Notations are made on Traffic Reports that samples are being sent to another laboratory.

A work-order number is assigned to a group of samples according to client and matrix not to exceed 20 samples received together or over a period of 7 days. (A 14 day receipt may be used, per client request.)

In the event that client samples are received by the laboratory unexpectedly, a Project Manager or sales person contacts the client documented on the sample **COC** by phone. During that call, the Project Manager or salesperson verifies the information documented on the COC, such as the project name, parameters to be tested, test methods to be used, client contact person, date the data report is due, and the data reporting requirements. The Project Manager documents the client information and project requirements in the LIMS and proceeds with sample login.

For samples hand-delivered after business hours, the actual date of sample receipt is recorded on the COC. The date and time of sample receipt and sample condition are recorded on the OTR. "Hand-Delivered" is recorded on the COC and OTRs.

5.3 Storage Conditions

The sample custodians and the supervisor of the Receiving department are responsible for the security of the sample storage units. Sample preparation and off-shift personnel also have access to the sample storage units. A roster is posted on each sample cold storage unit in the receiving department identifying personnel authorized to access samples. Samples are requested from the receiving department by lab staff by completing an internal COC form (Figure 5-14).

Under the NELAC standards, samples requiring thermal preservation are stored under refrigeration at ± 2 °C of the specified preservation temperature. For samples with a specified storage temperature of 4 °C, storage at a temperature above the freezing point of water to 6 °C is considered acceptable. Samples are segregated and stored in a manner that prevents cross contamination.

5.4 Chain of Custody

5.4.1 Sample Custody

COC records are used to document the collection of field samples and the transfer of the samples to the laboratory. The COC forms remain with the samples during transport or shipment. If shipping containers and/or individual sample containers are submitted with sample custody seals, and any seals are not intact, it is noted on the original COC. Internal COC records are used to document the storage, possession, transfer and disposal of samples, sub-samples, sample extracts and digestates with**in** the laboratory. The COC documents the identity of all individuals who physically handle samples and the time periods associated with the sample handling. Initials of individuals may be used since a cross-reference is maintained by QA that includes the initials and full names of all individuals signing COCs (except for work performed for the State of Florida, and other work when specified, where full signatures are required.) Any corrections to COC information must be made with indelible ink by using a single horizontal line to strike through the incorrect entry, and dating and initialing the correct entry written adjacent to it. Access to all samples is controlled. The laboratory area is maintained securely and is restricted to

authorized personnel only. The sample custodian relinquishes possession to the lab**oratory** staff.

Depending on client and regulatory requirements, COC**s** may originate at the time pre-cleaned sample containers are sent from **CompuChem** to the field, or when the sample containers are filled in the field and **placed** into the coolers. New Jersey and Florida are two states that require COC initiation in the laboratory with the loading of pre-cleaned empty containers into the cooler and before shipment to the field. A representative of the Receiving department is responsible for initiating a glassware release COC in such cases.

An internal COC form is generated to accompany the physical transfer of the sample (including its extract or digestate) through the lab (Figure 5-15). Internal COCs, sample preparation and analytical worksheets, and instrument run logs are used to document the individuals handling the samples at all times. When the analysis is complete, the sample is returned to the sample custodian for lo**ng**-term storage and disposal.

5.5 Sample Retention and Disposal

Unused raw samples are retained in a controlled temperature environment for 60 days after data submission to the client, or approximately 90 days after sample receipt. Extracts are stored refrigerated for one year after data submission. ILM05.4 requires a one year retention of metals digestates. Sample receipt and report dates are documented in the LIMS.

The sample custodian is responsible for purging raw samples from cold storage at the prescribed time. All samples, digestates, leachates, and extracts and other sample preparation products are disposed of in accordance with Federal and State laws and regulations. Sample identifications are recorded in designated Sample Disposal Logbooks at the time of purging (Figure 5-16.) Once segregated by the custodian, samples are processed for disposal by the hazardous waste technician. Client information is removed from bottles at the time of purging. If a sample is part of litigation, disposal of the physical sample occurs only with the concurrence of the client.

Figure 5-1
Chain of Custody

№ C05880

CompuChem
a division of Liberty Analytical Corp.

CHAIN OF CUSTODY

501 Madison Ave.
Cary, NC 27513
Phone: 919-379-4100 Fax 919-379-4040

Page ____ of ____

Courier
Airbill No.
Sampling Complete? Y or N

Client/Reporting Information	Project Information
Company Name	Project Name
Address	Sampling Location
City State Zip	Turnaround time
Project Contact	Batch QC or Project Specific? If Specific, which Sample ID?
Phone #	Are aqueous samples field filtered for metals? Y or N
Sampler's Name	Are high concentrations expected? Y or N? If yes, which ID(s)?

Requested Analysis (include method and bottle type)

Matrices
GW - Ground water
WW - Waste water
SW - Surface water
SO - Soil/Sediment
TB - Trip Blank
RI - Rinsate
WP - Wipe
O - Other

[redacted]	Field ID	Collection Date	Collection Time	Matrix	# of bottles	HCl	NaOH	HNO3	H2SO4	MEOH	Other	[redacted]

Lab Use Only		Comments
Sample Unpacked By:	Cyanide samples checked for sulfide & chlorine? Y or NA	
Sample Order Entry By:	625 & Phenol samples checked for chlorine? Y or NA	
Samples Received in Good Condition? Y or N	608 samples checked for pH between 5.0-9.0? Y or NA	
If no, explain:		

Sample Custody

Relinquished by:	Date/Time:	Received by:	Date/Time:
Relinquished by:	Date/Time:	Received by:	Date/Time:
Subcontact? Y or N If yes, where?	Custody Seal(s) intact? Y or N	On Ice? Y or N	Cooler Temp: °C

Samples stored 60 days after date report mailed at no extra charge.

White & Yellow copy to lab • Pink copy for customer

Figure 5-2
EPA Organic Receiving Log

Page ____of ____

CompuChem, a Division of Liberty Analytical Corp.- EPA ORGANIC RECEIVING LOG

Client / Account EPA	Rec'd Date	Comments:
Case No:	Courier	
Temp Blk in cooler? Yes / No	Airbill No.	
Temperature:		
Tags? Yes / No		
Custody Seals? Yes / No Intact? Yes / No		

COOLER REC'D BY:	REVIEWED BY:
Samples Logged in by:	SDG #:
Number of TR's?	Work Order No.:
Return Airbill in cooler? Yes / No	

No.	Sample No.	Client ID	QC	Matrix	Sample Date	Sample Time	T. VOC	VOC	VOC SIM	SVOC	SIM	PEST	AR	Amount/Containers
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														
11														
12														
13														
14														
15														
16														
17														
18														
19														
20														

OrgRec.log.doc-02/24/06

Figure 5-3
EPA Inorganic Receiving Log

Page ____of

CompuChem, a Division of Liberty Analytical Corp.- EPA INORGANIC RECEIVING LOG

Client / Account EPA	Rec'd Date	Comments:
Case No:	Courier	
Temp. Blk in cooler? Yes / No	Airbill No.	
Temperature:		
Tags? Yes / No		
Custody Seals? Yes / No Intact? Yes / No		

COOLER REC'D BY:	REVIEWED BY:
SAMPLE LOGIN:	SDG #:
Number of TR's?	Work Order No:
Return Airbill in Cooler? Yes / No	
Cyanide Samples checked for sulfide & chlorine? YES / NA	

No.	Sample No.	Client ID	QC	Matrix	Sample Date	Sample Time	Total Metals	Cyanide	Dissolved Metals	pH	Amount/Containers
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

InorgReclog.doc-03/01/05

Figure 5-4
Quality Assurance Notice

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

A chlorine and sulfide check was performed on the above cyanide sample

The results are checked below.

Chlorine was detected _____
Sulfide was detected _____

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

Dispose - client will resample _____

Supervisor Signature/ID _____/_____

Date _____

QAN-R-1
971022

Qanr1 – 10/22/97:llc

Figure 5-5
Quality Assurance Notice

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is _____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ Date _____

Preservation Type_____ Preservative Lot Number_____

Preserved By_____ Date_____

QAN-R-2
020529

QAN-R-2:052902:llc

Figure 5-6
Quality Assurance Notice

CompuChem
a division of Liberty Analytical Corporation

QUALITY ASSURANCE NOTICE

Client _____
Case # _____
Type of Analysis _____
Receipt Date

For some organic and/or inorganic determinations temperature preservation at 4 degrees Celsius is required for environmental samples during shipment to the laboratory and prior to analysis. A temperature tolerance range is generally allowed. Temperature of a representative sample from the shipping container is taken and recorded by the receiving clerk at the time of sample receipt. This temperature is representative of all samples contained in the cooler.

The EPA CLP program requires the laboratory make notification when the temperature exceeds 10° C.

The State of North Carolina requires that samples must be iced to above freezing but ≤ 6° C during shipment.

Notification to other clients is either client or project dependent.

Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

The temperature of this sample at the time of receipt was determined to be _____.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Hand Delivery/Received on ice _____

Analyze - qualify with notice _____

Dispose - client will resample _____

Supervisor Signature/ID _____/_____ Date _____

QAN-R-3
060308

Figure 5-7
Quality Assurance Notice

CompuChem
a division of Liberty Analytical Corporation

Quality Assurance Notice

CompuChem ID#_____
Client ID#_____
Case #_____
SDG# _____
Receipt Date_____
Method_____

A chlorine check was performed on the above phenol sample and was determined to be present. A member of CompuChem's Customer Service Department contacted the client. The Receiving Department was instructed as follows:

Analyze – Qualify with notice and address in narrative: _____
Dispose – Client will resample: _____

Supervisor Signature/ID _____/_____

Date _____

QAN-R-4
010702

Qanr4 – 07/2/01:dce

Figure 5-8
Quality Assurance Notice

CompuChem
A Division Of
Liberty Analytical Corp.

Quality Assurance Notice

Case # _____ SDG# _____
Receipt Date _____ Matrix _____

In the USEPA Contract Laboratory Program (CLP) "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration (Document Number SOM01.2),"directions are provided dealing with a temperature blank, termed the USEPA Cooler Temperature Indicator.

If a cooler temperature indicator bottle is not present in the cooler, the laboratory is required to contact the Sample Management Office (SMO), inform them of that fact and use an alternative means of determining the cooler temperature.

The following is a list of options employed by CompuChem to determine the cooler temperature. For the Case/ SDG presented above, the option(s) used have been indicated by a check mark.

Note: Any of the options performed are done so immediately after the cooler has been opened and the determination made that the cooler temperature indicator bottle is absent.

Water Samples
___An aliquot from a sample bottle designated for extractable organics is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after a 3-minute equilibration period. The contents of the disposable container are then properly discarded.

___A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

Soil Samples
___A calibrated IR gun is used, as indicated for water samples.

As required by the organic SOW, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative.

Signature_____
Date_____

QAN-R-5
011324

Qanr5 – 06/22/10:bm

Figure 5-9
Quality Assurance Notice

CompuChem
A Division Of
Liberty Analytical Corp.

Quality Assurance Notice

Case # _____ SDG# _____
Receipt Date _____ Matrix _____

In the USEPA Contract Laboratory Program (CLP) "Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration (Document Number ILM05.4),"directions are provided dealing with a temperature blank, termed the USEPA Cooler Temperature Indicator.

If a cooler temperature indicator bottle is not present in the cooler, the laboratory is required to contact the Sample Management Office (SMO), inform them of that fact and use an alternative means of determining the cooler temperature.

The following is a list of options employed by CompuChem to determine the cooler temperature. For the Case/ SDG presented above, the option(s) used have been indicated by a check mark.

Note: Any of the options performed are done so immediately after the cooler has been opened and the determination made that the cooler temperature indicator bottle is absent.

Water Samples
___ An aliquot from a sample bottle is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after a 3-minute equilibration period. The contents of the disposable container are then properly discarded.

___ A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

Soil Samples
___ A calibrated IR gun is used, as indicated for water samples.

As required by the inorganic SOW, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative.

Signature_____
Date_____

QAN-R-6
0511159

Qanr6 – 06/22/10:bm

Figure 5-10
Quality Assurance Notice

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is _____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

1. Client notified laboratory that data **would** be reported to the State of North Carolina. _____
2. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ Date _____

Preservation Type_____ Preservative Lot Number_____

Preserved By_____ Date_____

QAN-R-8
060126

QAN-R-8:012606:jad

Figure 5-11
Quality Assurance Notice

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

Client _____
Case # _____
Type of Analysis _____
Receipt Date _____

For some organic and/or inorganic determinations temperature preservation at 4° C is required for environmental samples during shipment to the laboratory and prior to analysis. A temperature tolerance range is generally allowed. Temperature of a representative sample from the shipping container is taken and recorded by the receiving clerk at the time of sample receipt. This temperature is representative of all samples contained in the cooler. The EPA CLP program requires the laboratory make notification when the temperature exceeds 10° C. The State of North Carolina requires that samples must be iced to above freezing but ≤ 6° C during shipment. Notification to other clients is either client or project dependent.

Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

The temperature of this sample at the time of receipt was determined to be _____.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Hand Delivery/Received on ice _____

Analyze - qualify with notice _____

1. Client notified laboratory that data **would** be reported to the State of North Carolina. _____
2. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Project Manager/ID _____/_____ Date _____

QAN-R-9
060309

qanr9 – 3/09/06:jad

Figure 5-12
Quality Assurance Notice

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

A chlorine and sulfide check was performed on the above cyanide sample

The results are checked below.

Chlorine was detected _____
Sulfide was detected _____

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

1. Client notified laboratory that data **would** be reported to the State of North Carolina. _____
2. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Project Manager/ID _____/_____ Date _____

QAN-R-7
060126

Qanr7 – 1/26/06:jad

Figure 5-13
Example Bottle **Label**

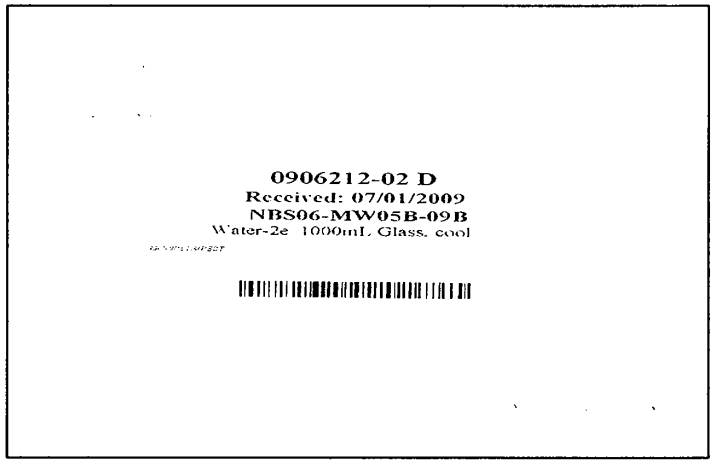

Figure 5-14
Internal Chain of Custody

CompuChem a Division of Liberty Analytical
INTERNAL CHAIN OF CUSTODY

Laboratory: _____ Requested By: _____

Water _____ Soil _____ P.E. _____ Date: _____ Shift: 1 2 Sat
circle one

EPA _____ Commercial _____ Time: _____

	SAMPLE NUMBER	RECEIPT DATE	PRES.	PARAMETER	RECEIVING USE ONLY BOTTLE NUMBER
1					of
2					of
3					of
4					of
5					of
6					of
7					of
8					of
9					of
10					of
11					of
12					of
13					of
14					of
15					of
16					of
17					of
18					of
19					of
20					of

Relinquished By: _____ Date: _____ Received By: _____ Date: _____ Reason: _____

Relinquished By: _____ Date: _____ Received By: _____ Date: _____ Reason: _____

Relinquished By: _____ Date: _____ Received By: _____ Date: _____ Reason: _____

Comments: _____

ic1 - 6/25/01:dce

Figure 5-15
Internal Extraction Chain of Custody

6/11/2009

Extractions Custody Sheet

Batch Status: Analysis

Lab Id	Client_Id	Received	Container	Extraction	Preservative	Matrix	Due Date	Cust Date

Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date

1

Figure 5-16
Example of a Sample Disposal Log

06/01/2010 through 06/30/2010 by DOVERC

Samples Ready for disposal; Biota, Oil, Soil, Soil Med, Tissue, Water, Wipe; 01_40mL VOA, cool, 01a_40mL VOA, cool, H2SO4, 01b_40mL VOA, cool, HCl, 01c_40mL VOA, cool, MeOH, 01d_40mL VOA, cool, Sodium Bisulfate, 01e_40mL VOA, cool, Sodium Thiosulfate, 01f_40ml VOA, Frozen, 100 mL Coliform

Disposed	Lab Number	Matrix	Container	Sample Name	Location	Hazard Info
-	1006152-01F	Water	3d_250mL Plastic, cool	PRGS-MH	Sample Receiving	
-	1006152-02E	Water	3h_250mL Plastic, cool, NaOH	TRENCH1	Sample Receiving	
-	1006152-02F	Water	3d_250mL Plastic, cool	TRENCH1	Sample Receiving	
-	1006152-03A	Water	2e_1000mL Glass, cool	TRENCH1-FD	Sample Receiving	
-	1006152-03B	Water	2e_1000mL Glass, cool	TRENCH1-FD	Sample Receiving	
-	1006152-04A	Water	2e_1000mL Glass, cool	TRENCH2	Sample Receiving	
-	1006152-04B	Water	2e_1000mL Glass, cool	TRENCH2	Sample Receiving	
-	1006152-04C	Water	2e_1000mL Glass, cool	TRENCH2	Sample Receiving	
-	1006152-04D	Water	2e_1000mL Glass, cool	TRENCH2	Sample Receiving	
-	1006153-01B	Water	01b_40mL VOA, cool, HCl	PRGS-MH	Sample Receiving	
-	1006153-01C	Water	01b_40mL VOA, cool, HCl	PRGS-MH	Sample Receiving	
-	1006153-02A	Water	01b_40mL VOA, cool, HCl	TRENCH1	Sample Receiving	
-	1006153-02B	Water	01b_40mL VOA, cool, HCl	TRENCH1	Sample Receiving	
-	1006153-04A	Water	01b_40mL VOA, cool, HCl	TRENCH2	Sample Receiving	
-	1006153-04B	Water	01b_40mL VOA, cool, HCl	TRENCH2	Sample Receiving	
-	1006153-04C	Water	01b_40mL VOA, cool, HCl	TRENCH2	Sample Receiving	
-	1006153-05A	Water	01b_40mL VOA, cool, HCl	TRIP BLANK	Sample Receiving	
-	1006154-01AC	Water	3d_250mL Plastic, cool	LS13-MW26S	Sample Receiving	
-	1006154-01AD	Water	3d_250mL Plastic, cool	LS13-MW26S	Sample Receiving	
-	1006154-01AE	Water	01a_40mL VOA, cool, H2SO4	LS13-MW26S	Sample Receiving	
-	1006154-01AF	Water	01a_40mL VOA, cool, H2SO4	LS13-MW26S	Sample Receiving	
-	1006154-01B	Water	01b_40mL VOA, cool, HCl	LS13-MW26S	Sample Receiving	
-	1006154-01C	Water	01b_40mL VOA, cool, HCl	LS13-MW26S	Sample Receiving	

Relinquished by coolers 1, 2A, 3, 5, 6, 8
Freezer 1, 3, 4 and ambient storage
Received by JAD Date 12/13/10 for disposal

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6.0 Reference Standard Preparation and Instrument Calibration

6.1 Standard Preparation and Storage

Reference standard solutions including those used for both sample preparation and instrument calibration are prepared by individuals within the various laboratory sections.

Standards are purchased as neat materials and concentrated solutions and are diluted to intermediate and working standards. The frequency of standard preparation varies with method requirements, stability and rate of consumption.

With the exception of organic CLP standards, standard preparations are verified with the analysis of initial calibration verification standards prepared using a different source or standard lot than that used to prepare the initial calibration.

Standard storage conditions vary depending on the method requirements and **vendor** recommendations. All standards used for volatile organic analyses are stored at –10 to –20 °C **in the volatiles storage freezer located in the volatiles laboratory**. Standards used for inorganic analyses are stored at room temperature **in the inorganics laboratory**.

6.2 Analytical Support Equipment

Analytical support equipment includes balances, ovens, refrigerators, freezers, water baths, thermometers, and volumetric dispensing devices (such as Eppendorf®, or dilutor/dispensing devices, if quantitative results are dependent on their accuracy). All support equipment is maintained in proper working order. The records of all activities, including service calls, are retained.

Oven temperatures are checked daily and balances are checked prior to use on each working day with NIST traceable references in the expected range of use and documented. Acceptance criteria are documented in the applicable logbook.

The temperatures of cold storage units containing samples (including sample extracts and digestates) are monitored daily. Cold storage units containing reference standards are monitored on the lab's normal days of operation. Monitoring is documented in the applicable logbook. **Minimum/Maximum thermometers are used to monitor each cold storage unit over weekends.**

Balances are calibrated semiannually by a qualified outside service organization over the entire range of use. Balance calibrations are verified each day of use with certified Class-S (NBS circular 547 or ASTM E617 Class 1 and 2) weights. **The weights are calibrated annually at the North Carolina Department of Agriculture and Consumer Affairs, Standards Division.**

All thermometers are calibrated annually against a NIST-certified thermometer. An infrared (IR) gun is used to measure sample temperatures upon receipt and is calibrated quarterly. **The NIST thermometer is calibrated annually at the North Carolina Department of Agriculture and Consumer Affairs, Standards Division.**

Automatic pipettes are calibrated by weighing three aliquots of water dispensed into a container and calculating the accuracy of the pipettes. **Verification of automatic pipettes used in the inorganics laboratory occurs monthly, while those in the organics laboratories occurs quarterly.**

If calibration criteria are not met, the equipment is removed from service until repaired or replaced.

6.3 Instrument Calibration

Instruments used in the analysis of samples must be calibrated at regular intervals as specified by the analytical test method and manufacturer's recommendations. The calibration procedures, frequency, acceptance criteria, corrective actions and reference standards are specified in the applicable analytical SOPs.

For mass spectral analysis, tuning is required prior to calibration. Instrument calibration procedures for organic analysis consist of an initial calibration, initial calibration verification, and continuing calibration verification.

Instrument calibration procedures for inorganic analysis consist of (a minimum of) an initial calibration, initial calibration verification, initial calibration verification blank, continuing calibration verification, and continuing calibration verification blank. Continuing calibration verification is performed at a minimum of every ten sample analyses.

Initial calibration verification standard solutions are prepared using reference materials from a source other than that used to prepare the initial calibration.

Information relative to instrument calibration is documented in instrument run logs. This information includes instrument identification, data file names, standard lot numbers, analysis dates, analysis times, analyst identification, and calibration status.

7.0 Test Methods and Standard Operating Procedures

CompuChem maintains Standard Operating Procedures (SOPs) that accurately reflect laboratory activities such as assessing data integrity, corrective actions, handling customer complaints, and all test methods. Procedures for test methods describing how the analyses are performed are specified in analytical SOPs and include sample preparation, cleanup and analysis. These are based on referenced methods published primarily by the EPA. Internally developed methods will be validated according to the EPA Performance-Based Measurement System, although none have been developed at this time. Method validation is discussed in Section 3.0. Controlled **hard** copies of SOPs are accessible to all personnel in each laboratory area, as well as electronically on the company's intranet site. Each SOP indicates the effective date, revision number, and signatures of the person who initiates the revision and those who approve the revision. **All personnel involved in fulfilling the requirements of a specific SOP are required to read and sign a confirmation form attesting to their understanding of the latest revision.**

SOPs are useful in training staff, in ensuring consistency among staff in performing procedures, and in saving time and effort. Any deviation from an established procedure is documented. The SOP collection is outlined in Table 7.1. A listing of methods performed at CompuChem and the corresponding SOPs is located in Section 2.0 of this manual.

7.1 SOPs for Test Methods

CompuChem's analytical procedures are divided into two categories, sample preparation procedures (SPPs) and instrument procedures (IPs). The Quality Assurance department controls SOP revision and distribution ensuring that only the most current approved procedures are documented and distributed for laboratory use. Documentation of procedures is critical to the assurance of data quality. These SOPs contain or reference to the following information:

- identification of the test method
- applicable matrix or matrices
- detection limit
- scope and application, including components to be analyzed
- summary of the method
- definitions
- interferences
- safety
- equipment and supplies
- reagents and standards
- sample preservation and storage
- quality control
- calibration and standardization
- procedure
- calculations
- method performance
- pollution prevention
- data assessment and acceptance criteria for quality control measures
- corrective actions for out-of-control data
- contingencies for handling out-of-control or unacceptable data
- waste management
- **additional** references
- tables, diagrams, and flowcharts,

7.2 Distribution Control of Standard Operating Procedures

SOPs are distributed by location and each lab area receives its own set of **applicable** SOPs. **A** complete set of original SOPs **and Table of Contents** is maintained in the Quality Assurance department. A master copy of the complete set resides on the intranet and is available to all personnel in a read-only electronic format.

7.3 Document Control of Standard Operating Procedures

The Quality Assurance department is responsible for formalizing (editing, assigning document control information), distributing, and tracking all SOPs. **Additionally, the QA Department ensures the SOPs are read by all appropriate staff members.**

7.3.1 Creating and Revising SOPs

When laboratory staff **or the QA Department** drafts a new SOP**,** or a revision to an existing SOP, the individual must sign a SOP Documentation Form (Figure 7-1) and obtain approval and the signature of the supervisor or a qualified second party. This form, along with a copy of the SOP, is submitted to the QA department. After the SOP has been word processed by QA and has had document control information added, it is then distributed to the area in which it is used. All appropriate lab personnel read the SOP and sign the SOP Confirmation Form (Figure 7-2). The date that the SOP is distributed for use becomes the effective date of the SOP. The original, master, and controlled copies are generated and distributed to the appropriate locations **including electronically on-line**. The SOP Confirmation Form is **scanned and** archived by QA.

7.3.2 Archiving SOPs

When a procedure becomes obsolete, the request is made to remove it from the SOP collection. The SOP is removed from the active SOP table of contents to the Archival SOP table. The SOP is then removed from each collection. Archived SOPs are maintained indefinitely electronically.

7.3.3 SOPs as Training Tools

All new employees are required to read SOPs pertinent to their job functions as part of their training. A SOP Signature Form is completed and signed by the employee and his/her immediate supervisor/manager to document that the employee has read the SOPs pertinent to his/her job function. Supervisors document various training aspects using checklists and most include a list of the required SOPs. **A copy of the signature form(s) are maintained in an individual's training file.**

7.4 Document Control of the Quality Manual

The Quality Manual is revised and edited solely by the QA department. Revision numbers are provided with each update, and each section may be updated independently of others. There are controlled copies of the Quality Manual available to all laboratory

staff. Quality Manuals that are sent off-site are for informational purposes only and are not controlled documents unless requested by the client. In that event, the issue is assigned a tracking number and a copy of the Quality Manual is sent to the recipient with each revision. On an annual basis, the Quality Manual is distributed laboratory-wide for all staff to read. Signature sheets are maintained to document this review. **In general, the QA manual is updated yearly.**

7.5 Document Control of Laboratory Logbooks and Sample Preparation Worksheets

The QA department has primary responsibility for document and distribution control of over **200** logbooks used in the laboratory. To ensure document uniformity and compliance with NELAC standards, the U.S. EPA, good laboratory practices, and other certifying agency protocol, specific document control procedures are used for all laboratory logbooks and run logs.

Document control header information appears along the top of each logbook page and identifies the laboratory or administrative area, the logbook, and the issue number. Each logbook issue is consecutively paginated and permanently bound.

A logbook is developed cooperatively by the area manager and the QA department. The requestor submits a completed Logbook Request Form to the QA department. They confer to design a prototype logbook page that meets the needs of the laboratory and contains the key elements required by the QA department. These elements are:

- **Unique logbook identifier**
- The identity of the task
- The name CompuChem, a Division of Liberty Analytical Corporation
- A "Reviewed by" signature field
- A date of review field
- Any applicable measurement acceptance ranges with instructions for reporting out-of-range readings
- A corrective action statement
- Model specifications for equipment
- Standard lot number fields

An alphanumeric identifier is assigned to each logbook. The laboratory notifies QA when the logbook is ready for archival. Before turning over the logbook issue to QA, the manager of the area in which the logbook is used or a designee must review the contents of the logbook and sign the Logbook Authorization form, which is the last bound page.

The laboratory staff must complete the logbooks with the proper quality details. Any changes made must not obliterate or overwrite the original entry. Personnel make a single strike through the entry and initial and date the correct entry that is made adjacent to the original. Correction tapes and fluids are not allowed when completing laboratory logbooks or other records.

When completed, the laboratory returns the logbook to QA for archival. The issue is then logged into the Logbook Archival **Excel spreadsheet** and assigned to a unique **storage** box number. The logbook is then **maintained in a waterproof, ziplock bag, and placed** into the appropriate box for archival.

The QA department also **oversees** laboratory sample preparation worksheets**,** which include the same elements as logbooks. Logbook pages are included as attachments in SOPs. These are not bound nor paginated for ease in photocopying for data reports. When completed the sample preparation staff return the worksheets to QA for archival. The worksheets are then paginated and logged into the Worksheet Archival Log. The worksheets are assigned to a unique box number. The worksheets are then placed into the appropriate box for archival.

7.6 Records Retention and Purging

Archived laboratory records are stored on site **in a secured location** for a period of five years, or longer per client request. (Data retention is 10 years for the Ohio Voluntary Action Program.) **Purged data are processed by a shredding company specializing in destroying confidential material.**

7.7 Method Modifications

7.7.1 Total Organic Carbon in Soil -- Method 9060

The water method is adapted for soil matrices. Soil samples are analyzed using a boat sampler attachment in which the samples are introduced into a 900°C combustion zone, where all carbonaceous matter is oxidized to carbon dioxide. The carbon dioxide level is then determined using the non-dispersive infrared analyzer.

7.7.2 Aqueous Metals Digestion – Method 3010A

The block digestion technique utilizing an aqueous 50 ml sample aliquot, adjusted to a final volume of 50 ml, is used in the preparation procedure.

7.7.3 pH – Method 4500 H+ B

Samples should be analyzed immediately. Since the sample pH is measured in the laboratory and not in the field, the method holding time cannot be met.

Figure 7-1

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure _____ outdated procedure (archive) _____

- Procedure Code:_____ SOP Section #: _____ Revision #:_____

SOP Title: Effective date: (QA fills in)

_____ _____

_____

_____

- Procedure prepared by: Date:

_____ _____

- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date:

_____ _____

- Reason for change:_____

_____

- This procedure meets the requirements of the following approved method references:

_____

_____

_____

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date:_____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

5sopdocform2-1/19/07: tr

Figure 7-2

CompuChem
A Division Of
Liberty Analytical Corp.

SOP Confirmation Form

A new or revised version of the SOP documented below has been placed in your SOP collection. Take the time to read the procedure. For revised SOPs, new text is in **bold** print. After reading the new or revised procedure, sign and date this form in the spaces provided below. After all personnel that perform the procedure have signed this form, please return it to the Quality Assurance Department.

_____ _____
Manager/Supervisor Signature Date

SOP Title: _____

_____

Section # _____

Rev. # _____ Area/Lab_____

Effective Date_____
(QA fills in)

Department Signatures

I acknowledge review of the SOP documented above and agree to adhere to the procedures outlined in that document.

Printed Name	Signature	Date

sopcf1 r4.doc.doc
3/19/10:jad

Table 7.1

SOP OUTLINE

ANALYTICAL STANDARD OPERATING PROCEDURES

1.0 Purgeable Organics
- 1.1 VOA GC
- 1.2 VOA GC/MS CLP
- 1.3 VOA GC/MS Non-CLP
- 1.4 VOA GC/MS Miscellaneous

2.0 Extractable Organics
- 2.1 GC CLP
- 2.2 GC Non-CLP
- 2.3 HPLC
- 2.4 SV GC/MS CLP
- 2.5 SV GC/MS Non-CLP
- 2.6 Clean-up Procedures
- 2.7 Leachate Generation Procedures
- 2.8 Miscellaneous Preparation Procedures

3.0 Inorganics
- 3.1 Metals CLP
- 3.2 Metals Non-CLP
- 3.3 Mercury
- 3.4 Cyanide
- 3.5 Wet Chemistry
- 3.6 Organic Characterization

NON-ANALYTICAL STANDARD OPERATING PROCEDURES

4.0 Sample Control
5.0 Customer Services
6.0 Sample Scheduling
8.0 Organic Sample Preparation Laboratory
9.0 Inorganic Sample Preparation Laboratory
10.0 Glassware Preparation Room

Table 7.1 (continued)

OUTLINE OF NON-ANALYTICAL STANDARD OPERATING PROCEDURES

- 11.0 Instrument Laboratories
  - 11.1 Purgeable Organics
  - 11.2 Extractable Organics
    - 11.2.1 GC
    - 11.2.2 SV GC/MS
  - 11.3 Inorganics
    - 11.3.1 Metals
    - 11.3.2 Wet Chemistry & Organic Characterization
- 12.0 Hazardous Waste Management & Safety
- 13.0 Quality Control
- 14.0 Data Review
  - 14.1 Purgeables
    - 14.1.1 VOA GC and GC/MS
  - 14.2 Extractable Organics
    - 14.2.1 GC
    - 14.2.2 SV GCMS
  - 14.3 Inorganics
    - 14.3.1 Metals
    - 14.3.2 Wet Chemistry & Organic Characterization
  - 14.4 Data Release, Storage &Archival
- 15.0 Computer Operations
- 16.0 Human Resources
- 17.0 Quality Assurance

8.0 Data Quality Indicators

The data acquired from quality control (QC) procedures are used to estimate the quality of the analytical data, determine the need for corrective action in response to identified deficiencies, and to assess the effectiveness of the corrective actions implemented. Each analytical SOP includes a QC section that documents the specific QC requirements for that procedure.

8.1 Quality Control Samples

8.1.1 Blanks

8.1.1.1 Method Blanks

Method blanks are prepared and analyzed at a rate of one per **client** sample batch not to exceed 20 field samples, of the same matrix and test method. Method blanks are used to **assess** the batch for possible contamination during sample processing. Samples associated with a blank that does not meet quality control acceptance criteria are re-prepared and analyzed.

8.1.1.2 Storage Blanks

Storage blanks are generated each day CLP SDGs are received for volatile analysis. These blanks are stored with each SDG and are analyzed after all samples in the SDG have been analyzed. The results of the storage blank indicate whether contamination occurred during sample storage. While storage blanks are required for CLP SDGs, they may also be associated with non-EPA client samples stored in the sample cold storage unit.

8.1.1.3 Trip Blanks

For volatile analyses, a blank is placed in each cooler that is delivered to the client for sample collection. The blank remains with the samples during transport to the lab for analysis. The results of the trip blank indicate whether contamination occurred during sample transport.

8.1.2 Laboratory Control Samples

A Laboratory Control Sample (LCS) is prepared and analyzed with each sample batch not to exceed 20 field samples. An LCS is not required for those analytes **when** spiking solutions are not available such as pH. The LCS is used to assess whether the total analytical system is in control. The results of the LCS determine batch acceptance. Samples associated with an out of control LCS are re-prepared and analyzed.

8.1.3 Matrix Spikes

Matrix spikes are prepared and analyzed at the frequency required by the client, **Quality Assurance Project Plan (**QAPP**)**, or EPA region. The compounds

spiked should be specified in the client QAPP or EPA CLP SOW. Matrix spikes are used to assess the effect the sample matrix has on the precision and accuracy of the test method. The results of the matrix spikes are expressed in percent recovery and relative percent difference between duplicate matrix spikes. The results of the matrix spikes do not determine the acceptance of the sample batch.

8.1.4 Sample Duplicates

Sample duplicates are prepared and analyzed at the frequency required by the client, QAPP, or EPA region. The sample duplicate is used to assess the precision of the test method. The results of the sample duplicate do not determine acceptance of the batch. Matrix spike duplicates may be used as sample duplicates.

8.2 Surrogates and DMCs

Surrogates and DMCs are added to all samples, standards, and blanks for all organic analyses. The surrogate recoveries are used to assess preparation/extraction efficiency. Surrogate percent recovery acceptance criteria used by the laboratory are presented in the test method or are derived statistically by the laboratory. Surrogate failures may indicate poor extraction efficiency or matrix interferences. Field and QC samples that do not meet surrogate acceptance criteria are reanalyzed or re-prepared and analyzed.

8.3 Internal Standards

Internal standard solutions are added to all calibration standards and field and QC samples for mass spectral analyses. Internal standards are used to quantitate results. The laboratory's acceptance criteria for internal standard responses are those specified in the applicable analytical method.

8.4 Spiking Solutions

The compositions of spiking solutions used vary according to method and client requirements. Typically the solutions will contain all compounds of interest in a project and that are commercially available. For some clients (EPA Region), a representative subset of compounds may be used. Spike percent recovery criteria are presented in the test method or are derived statistically by the laboratory.

8.5 Calibration Verification

Initial instrument calibration verification is performed using a reference standard solution acquired from a second manufacturer or lot **other** than the solution used for the initial calibration. Initial calibration verification is required for inorganic methods and organic non-CLP methods. The initial calibration verification (ICV) standard is analyzed after the initial calibration and generally before any samples are analyzed.

When an initial calibration is not performed on the day of sample analysis, the initial calibration is verified by the analysis of the continuing calibration verification (CCV)

standard. If the solution used for the CCV standard is from a second manufacturer or lot, analysis of an ICV is not required.

8.6 Selectivity

Selectivity is evaluated by using the checks established within the analytical methods.

GC/MS and ICP-MS instrumentation is tuned to meet method requirements and these acceptance criteria are documented in SOPs.

Retention time window are established for GC and HPLC analyses and second column confirmation is performed. Acceptance criteria for retention time windows are documented in **the method specific** SOPs.

Inter-element interference checks are analyzed for ICP and ICP-MS methods. Acceptance criteria are documented in **the method specific** SOP.

8.7 Quality Control Sample Management

The analytical and quality control requirements for each sample are documented in the LIMS. The LIMS uses a **structured** system of test codes to schedule the appropriate analytical procedures and the QC samples required for each **unique** work-order or sample delivery group (SDG). The test codes have the analytical methods associated with them and are assigned to each sample upon receipt. Product codes may be defined to allow for specific requirements of a client or Quality Assurance Project Plan (QAPP).

8.7.1 Batching

8.7.1.1 Preparation Batch

A preparation batch is a group of no more than 20 field samples prepared (extracted or digested) together with a method blank. A Laboratory Control Sample (LCS) is required for **all** non-CLP samples.

8.7.1.2 Analytical Batch

An analytical batch is a group of no more to 20 samples, sample extracts, or sample digestates (including QC samples) that are analyzed together on the same instrument. For analyses that have no preparation step (volatiles), the method blank, LCS, matrix spike and matrix spike duplicate are included in the 20 samples.

9.0 Data Reduction, Review and Reporting

Computer software used for data reduction is validated before use. A test set of data, with manually calculated or previously validated results, is used to validate the new or revised software routine. Procedures for software validation are documented in SOPs. All data used in producing the final report are retained either as a hard copy or electronic file to insure that all results can be reproduced. These data include all instrument tune files, calibration files, sample files, QC sample files, standard preparation records, and sample preparation records. The following is a summary of the data review process. The analyst review responsibilities may vary slightly between the individual laboratory groups.

9.1 Data Review for Organic Analyses

Data collected by GC, GC/MS, and HPLC instrumentation software are transferred to Hewlett Packard UNIX servers for processing and review. The data **are** accessed by analysts and data reviewers through their personal intranet log-on **and through state-of-the-art data processing software**.

All data are subjected to a three-tier review before being reported to the client.

The analyst documents all analyses on the instrument run log. The analyst insures that all tune, calibration, method blank, QC sample and field sample data meet acceptance criteria. The analyst prints hard copies of the tune and calibration data generated by the instrument. The analyst signs (initials) and dates any manual integration he, or she, performed.

The data reviewer insures that all weights, volumes, and percent moistures are transcribed accurately into the raw data for calculation purposes. The data reviewer insures that the proper preparation and analysis methods have been used and that all the compounds of interest have been included in the analysis. The data reviewer insures that all tune, calibration, method blank, QC sample and field sample data meet acceptance criteria. The data reviewer assesses the spectra of target and tentatively identified compounds. The data reviewer prints hard copies for all method blanks, QC samples and field samples included in the work-order/SDG. The data reviewer signs (initials) and dates the hard copy reports as well as any manual integration he or she performed.

Manual integrations are performed according to the procedures outlined in SOP 13.18 "Manual Chromatographic Peak Integration Procedures".

A senior level staff member or a supervisor performs the final review. The final reviewer insures that all weights, volumes, and percent moistures are transcribed accurately into the raw data for calculation purposes. The final reviewer prints hard copies of all report forms and generates any electronic data deliverable (EDD). The final reviewer summarizes the data in the work-order/SDG narrative documenting any anomalies in sample collection, receipt and analysis. **The finalized hard copy is then ready for scanning, paginating, and delivery to the client as specified by their SOW requirements.**

9.2 Data Review for Inorganic Analyses

Metals data collected on the ICP and ICP-MS instrumentation software are transferred to **the** LIMS for review and reporting. Wet Chemistry data collected on the Lachat instruments are transferred to the LIMS for review and reporting. Wet Chemistry data not collected on the Lachat are manually entered into Excel spreadsheets by the analyst. The information entered into these spreadsheets is transferred to the LIMS for reporting.

Data for inorganic parameters **are** subjected to a two-tier review before being reported to the client.

The analyst documents all analyses on the instrument run log. The analyst insures that all weights, volumes, and percent solids are transcribed accurately into the raw data for calculation purposes. The analyst insures that all calibration, method blank, QC sample and field sample acceptance criteria are met. The analyst insures that all raw data transcribed into spreadsheet **are** accurate.

A senior level staff member or a supervisor performs the final review. The final reviewer insures that all weights, volumes, and percent solids are transcribed accurately into the raw data for calculation purposes. The final reviewer insures that all calibration, method blank, QC sample and field sample acceptance criteria are met. The final reviewer summarizes the data in the work-order/SDG narrative, documenting any anomalies in sample collection, receipt and analysis. **The final reviewer prints all reporting forms and generates the EDD, and prepares the hard copy for scanning, paginating, and delivery to the client as specified by their SOW requirements.**

9.3 Data Reporting

It is CompuChem's policy to report data that meet all quality control criteria, to the extent possible. If deficiencies are identified at any point in the review process, corrective action is taken. Documentation of any corrective action or data qualification is done by the final reviewer in the narrative.

Each fraction is reviewed separately and the individual reports that make up the final report are produced. The final report is scanned, paginated, saved in a portable document format (pdf), printed and the pdf archived. The printed final report **and/or CD** is sent to the client.

The final report format and detail are tailored to the specific client needs.

Figure 9-1 shows how data flow through the laboratory, from sample receipt to final report.

9.4 Confidentiality

Results of sample analyses and associated raw data are reported to the customer requesting the analyses. Confidentiality is preserved to the extent possible when clients

require transmission of test results via telephone, fax, e-mail**, or data upload through an internet portal**.

A version of the following statement accompanies out-going fax and electronic mail correspondence:

> "This e-mail and any information contained or attached to this e-mail are privileged, confidential and proprietary information intended only/solely for the individual or entity to which it is addressed. It is a PRIVATE e-mail message and the sender does not waive any related rights and obligations. Kindly notify the sender immediately by e-mail if you have received this e-mail in error. You are notified that disclosing, copying, distributing or taking any action in reliance on contents of the information is strictly prohibited."

Access to data reports and other laboratory records is available to external auditors (representatives of accrediting bodies and agencies) during on-site assessment. Data reports and other laboratory records are provided **confidentially and anonymously** upon request to auditors off-site to support the laboratory's certifications.

9.5 Data Security

The Information Technology department assures the security of the computer systems by assigning log-on accounts and individual passwords for individuals to access data. The user is restricted to certain menu options through the log-on account, and only authorized staff has access to editing capabilities. A software product controls menus and uses local attributes consisting of specific options available to authorized users. These attributes are created and maintained by the Information Technology staff. Access levels vary and depend on the user job functions. **Individual** log-on passwords are required to be changed every six months **and** workstations are set up to lock after 10 minutes of inactivity.

The data servers located in the Information Technology department are linked to an Uninterruptible Power Supply (UPS). All data saved on the data servers and personal computers are backed up at varying frequencies. The HP UNIX servers are backed up incrementally on a nightly basis and fully once each week. Selected directories on personal computers are backed up monthly. Archiving procedures are performed in compliance with Information Technology SOPs. The archive storage media are stored in a restricted area within the facility, secured by locked doors, with limited access. Access is allowed only by means of an electronic security access key. The storage area is air-conditioned and kept free of debris. An additional copy of the archive storage media is maintained off-site.

9.6 Hard Copy Data Retention

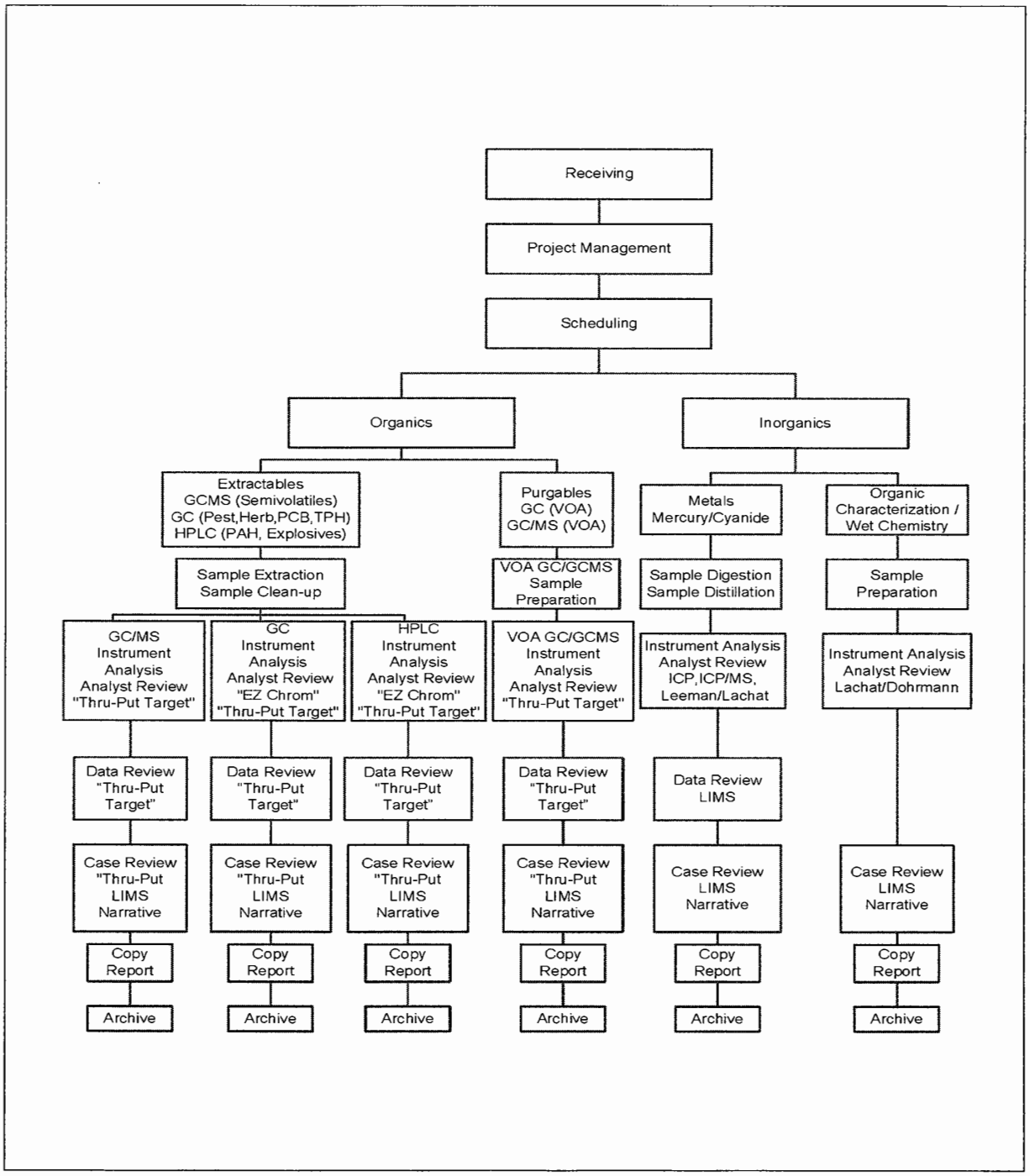
The laboratory scans the hard copy data, paginates it, and sends it to the client. Only the scanned copy is retained on **a** database at the laboratory, unless the client requires otherwise. If the client requires that a hard copy of the final report and associated data

be retained by the laboratory, the data are boxed and placed in a secure room within the facility. The laboratory retains the data for 5 years, or as specified by the client.

9.7 Electronic Data Retention

Archived data are stored for a minimum of five years, except for EPA who requires one year **retention**. Additional storage time may also be requested by the client for certain projects or required under certain programs such as the Ohio Voluntary Action Program (VAP) **or certain commercial clients who** require a data retention time of at least 10 years. It is the responsibility of the laboratory project manager to oversee the extended retention of project files. **An** auxiliary data field in the LIMS is able to record data retention time requirements for each client and to assist in tracking extended retention times. For the Ohio VAP, the laboratory must notify the agency by certified mail when the 10-year retention period has expired and retain the data until directed in writing by the agency whether they will or will not retain the data.

Figure 9-1
Sample Data Flow Chart



10.0 Performance and System Audits and Frequency

10.1 Quality Assurance Audit Unit

The QA department staff consists of senior scientists with Bachelor of Science or Bachelor of Arts degrees **with an emphasis on chemistry and a combination of other biological or environmental academic disciplines**, ten or more years of environmental analytical laboratory experience, and at least five years of experience using laboratory QA/QC techniques.

The QA **staff** conduct internal system audits. The auditor applies specific audit methods, evaluates audit findings, and reports these findings to laboratory management.

10.2 Systems Audits

A systems audit is an on-site inspection or self-assessment of the laboratory's control systems. System audits are performed both by internal and external auditors. Clients and federal and state certifying agencies perform external system audits. A system audit is performed to qualitatively assess the laboratory's control systems and is intended to provide evidence of the laboratory's competence.

The objectives of a systems audit include ensuring that:

- management is committed to creating a work environment dedicated to quality and that a structured management system is in place to support an effective QA program,
- the QA program is documented and implemented to assess work to ensure **ethical,** technical, administrative, and quality objectives are met,
- personnel are adequately trained and qualified to do their jobs,
- procured items and services meet established requirements and perform adequately,
- procedures are established and maintained for the preparation, review, revision, and issuance of laboratory documents, including SOPs, logbooks, and data reports
- records are retained for evidentiary purposes,
- computer hardware is maintained to support data processing and reporting software
- computer software is developed, documented, validated and any changes are controlled and documented
- **equipment and instrumentation is maintained and serviced at appropriate intervals**
- work performed complies with established documented procedures**,** as well as safety policies, and
- procedures are established for detecting and preventing quality problems and for ensuring quality improvement.

10.2.1 Internal Systems Audits

Internal systems audits are used to verify that laboratory operations continue to comply with the requirements of the quality system. Systems audits include a review of laboratory documentation on sample receiving, sample log-in, sample storage, chain-of-custody procedures, sample preparation, sample analysis, instrument operating records, etc. While performance audits are a quantitative appraisal, system audits are more qualitative in nature, intended to provide evidence of the laboratory's competence.

Upon completion of an internal system audit, the auditor discusses **their** findings with the responsible manager or supervisor. The auditor summarizes **their** findings in an audit report that is distributed to laboratory management. A Corrective Action Report (CAR) is issued for each finding**, if warranted**. The responsible manager corrects the deficiency, documents the correction in the CAR, and returns the completed CAR to the auditor. Completed CARs are retained in the QA department. Implementation of the documented corrective actions is verified by subsequent follow-up audits. If an audit finding were to cast doubt on the correctness or validity of the laboratory's results, an immediate corrective action would be initiated and any client whose work may have been affected would be notified.

Preventative Action Reports (PARs) can be initiated by any individual and submitted to their supervisor or QA. PARs are similar to CARs, but function to address potential non-conformances.

A full internal systems audit is performed semi-annually. The following components comprise a complete system audit. Below is a summary of the audit activities performed by QA staff. Figures 10-1 through 10-5 are example checklists used to document these activities.

10.2.1.1 **Documentation and Record Keeping** – Review logbooks that document refrigerator temperature, instrument analysis, balance calibration, and standard preparation. Verify that records are complete, that documented observations are current, that any errors are corrected properly, and that completed pages and logbooks were peer reviewed as evidenced by a review signature. **For organics, supervisor review and signature is required for every twenty pages of a run log.**

10.2.1.2 **Sample storage/Chain of Custody** – Review temperature logs for refrigerators that store samples and sample extracts. Verify that samples are stored and controlled properly and that COC procedures are followed through documented transfers.

10.2.1.3 **SOP compliance** – Observe a procedure being performed by an analyst to verify that the procedure being performed follows the written SOP. Also verify that the written SOP is compliant with the referenced methods.

10.2.1.4 **Reagent (solvent and chemical) storage and control** – Observe and verify the proper storage conditions of reagents and solvents. Verify that proper control documentation is written on

the container label such as the receipt date, expiration date, **and standard lot identifier**.

10 2.1.5 **Standard storage and control** – Observe and verify the proper storage conditions of reference standards. Verify that proper control documentation is written on the container label such as the standard lot number and expiration date. Review standard logbooks to verify that standard preparations are properly documented and traceable to primary standards. Also verifies that standards are traceable to NIST sources.

10.2.2 **QC test sample analyses** – Review of data associated with the analysis of new solvent lots.

10.2.3 **Data audits** – QA staff routinely audit all data associated with the analyses of proficiency test samples. EPA CLP data are reviewed routinely by the Vice President and General Manager as a result of EPA CCS reports. During these audits, QA staff verify the data for accuracy, completeness, and test method and program compliance. Software data reduction routines are verified during data audits by performing manual calculations or by reviewing the calculation verification provided with the data report. Hard copy to diskette results may be compared in the course of a data audit, but are typically verified by automated software routines.

10.2.4 **ThruPut systems Audit Trail** – The audit trail in ThruPut systems is checked semi-annually to verify changes to the processed files are correct, documented, allowed and performed by authorized individuals, **and that manual integrations are initialed and dated by the data reviewer**.

10.2.5 **Corrective Action Report (CAR) follow-up** – Verifies that corrective actions have taken place, are still in effect, and that recurrence is controlled.

10.2.6 **Facilities maintenance** – Verifies that facilities and equipment are adequate and properly maintained and that laboratory areas are free from interference or contaminants **and that safety measures are being observed and compliant**.

10.2.7 **Subcontract laboratory audits** – In lieu of on-site audits, a paper audit may be performed involving a review of the quality assurance plan, SOPs, data reports, and PE study results. On-site audits can be performed upon client request, but no routine audit is performed by our lab.

10.2.8 **Employee training file audit** – Verifies that training records are current and that personnel meet the **educational and experience** requirements stated in the current USEPA CLP SOW, as well as those specified by individual states.

10.2.9 **External Systems Audits**

CompuChem's systems are also inspected extensively by external agencies, contractors, and third parties. The laboratory's primary NELAC accrediting authority conducts an assessment of the laboratory every two years. As a laboratory in the USEPA CLP, CompuChem is audited annually by representatives of the EPA. Many clients conduct inspections or **use** third party

QA auditors to inspect the laboratory before start-up and during the course of larger, more critical, or sensitive projects.

CompuChem prefers a two-week notification before a scheduled audit to ensure that management and QA staff are available. However, an external audit may be conducted (announced or unannounced) at any time during normal business hours. To protect client confidentiality, some documents (particularly those identifying clients, sites, or projects) will not be made available for inspection except to those directly involved in such projects or authorized state or federal officials or authorized third parties.

Any deficiencies or non-conformances observed by the auditors are included in an audit report. Corrective action reports are initiated as needed in response to the audit findings. Once the corrective action responses are completed, a formal response is compiled by a QA staff member and submitted to the external agency by the required due date. Copies of the response are circulated to laboratory management. Follow-ups are performed to ensure implementation of stated corrective actions.

10.3 Proficiency Test(PT) and Performance Evaluation (PE) Samples

Laboratory performance is evaluated using proficiency test and performance evaluation samples.

CompuChem analyzes water pollution (WP), solid matrix (SM)**, and underground storage tank (UST)** proficiency test (PT) samples semiannually in support of its NELAC accreditation. The laboratory analyzes single blind performance evaluation (PE) samples quarterly in support of its participation in the EPA's contract laboratory program.

As part of its data audit function, the QA department audits all PT and PE sample data before results are submitted. PT and PE sample results are summarized in memoranda that are distributed to laboratory management. **CARs are initiated for root cause of failed results.**

10.4 Management Review

The results of the annual internal systems audit performed by the QA staff are reported to the laboratory management. Corrective action reports are distributed to the managers documenting any deficiencies. Corrective actions for these deficiencies are documented on the reports and returned to the QA department. **CARs are tracked for trending purposes.**

The QA Manager **and QA Specialist** report on activities done in support and development of the laboratory's quality system in writing on a monthly basis to the Vice President and General Manager.

The Vice President and General Manager also writes a report on activities done in support of the laboratory's quality system and discusses quality system issues with senior management during staff meetings.

Figure 10-1

CompuChem
A Subsidiary of
Liberty Analytical Corp

SYSTEM AUDIT CHECKLIST
Sample Storage/Chain of Custody

Auditor_____Date_____Department_____

1. Are refrigerators well organized?	Yes	No
2. Is ID Label on door?	Yes	No
3. For SOM01.2, is the SOP posted on the door?	Yes	No
4. Is temperature range posted?	Yes	No
5. Is corrective action stated in logbook?	Yes	No
6. Are temperatures within range?	Yes	No
7. Are standards and samples stored separately?	Yes	No
8. Are logbook errors corrected properly?	Yes	No
9. Has the supervisor reviewed and signed off?	Yes	No
10. Are logbooks completed to the point of inspection?	Yes	No
11. Are samples under proper chain of custody?	Yes	No
12. Is chain of custody record completed?	Yes	No
13. Are samples left out of refrigeration more than 2 hrs since receipt?	Yes	No

Comments:

1. _____
2. _____
3. _____
4. _____
5. _____
6. _____
7. _____
8. _____
9. _____
10. _____
11. _____
12. _____

SAC1:1/10/11: bm

Figure 10-2

CompuChem
A Division Of
Liberty Analytical Corp.

DOCUMENTATION AND RECORD KEEPING AUDIT CompuChem

Auditor Date Laboratory

Logbook Name and ID #	Page # or Date	Detailed Documentation? Yes/No	Corrective Action Documented? Yes/No	Proper Error Correction? Yes/No	Page z'd-out? Yes/No	Review sign-off? Yes/No	Comments

SAC2:2/11/10:jr

Figure 10-3

CompuChem
A Division Of
Liberty Analytical Corp

SYSTEM AUDIT CHECKLIST
SOP Compliance

Auditor_____Date_____Department_____
SOP Title:_____
Instrument Procedure No._____ or Sample Preparation Procedure No. _____

1. Is the procedure performed according to the SOP?	Yes	No
2. Is the SOP compliant with referenced methods?	Yes	No
3. Does the SOP require revision?	Yes	No
4. Is corrective action necessary?	Yes	No
5. Is there documentation of training for this chemist and procedure?	Yes	No

Comments
1. _____
2. _____
3. _____
4. _____
5. _____

Corrective Action Follow-up
1. _____
2. _____
3. _____
4. _____
5. _____

SAC1:1/10/11: bm

Figure 10-4

CompuChem
A Division Of
Liberty Analytical Corp.

SYSTEM AUDIT CHECKLIST

Standard Storage and Control

Auditor_____Date_____Department_____

1. Is the standards refrigerator/freezer well organized?	Yes	No
2. Is ID Label on door?	Yes	No
3. Is temperature range posted?	Yes	No
4. Is corrective action stated in logbook?	Yes	No
5. Are temperatures within range?	Yes	No
6. Are standards and samples separated?	Yes	No
7. Are logbook errors corrected properly?	Yes	No
8. Has the supervisor reviewed and signed off?	Yes	No
9. Are logbooks completed to the point of inspection?	Yes	No
10. Are shelves or racks clearly labeled and are active standards segregated from expired/unapproved/special standards?	Yes	No
11. Are standards left out of refrigeration more than 2 hrs?	Yes	No

List below standard lot numbers currently in use for traceability to the standards preparation logbooks.

Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____

List below any standard Ids and lot numbers of expired standards, if any, stored with current standards.

Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____
Standard ID _____ Lot Number _____ Expiration date _____

Comments

1. _____
2. _____
3. _____

SAC2:2/11/10:jr

Figure 10-5

CompuChem
A Division Of
Liberty Analytical Corp

SYSTEM AUDIT CHECKLIST

Reagent (Solvent and Chemical) Storage and Control

Auditor_____Date_____Department_____

1. Are chemicals stored properly (ether in flammables cabinet, etc.)?	Yes	No
2. Are bottles labeled with date received, opened, and initials?	Yes	No
3. Are expiration dates determined (i.e., 5 yr. from receipt or manufacturer recommendation)?	Yes	No
4. Are any beyond the expiration period?	Yes	No
5. Are materials logged into the materials receipt log (inorganics)?	Yes	No
6. Are materials traceable to the receipt log (inorganics)?	Yes	No
7. Are all bottles labeled with the identity of the contents?	Yes	No
8. Are reagents being made at the required frequency?	Yes	No
9. Are proper safety equipment worn when handling reagents?	Yes	No
10. Is the area clean of debris or spills?	Yes	No

List below reagent lot numbers currently found to be in use.

Reagent ID _____ Lot Number _____ Expiration date _____
Reagent ID _____ Lot Number _____ Expiration date _____
Reagent ID _____ Lot Number _____ Expiration date _____
Reagent ID _____ Lot Number _____ Expiration date _____
Reagent ID _____ Lot Number _____ Expiration date _____

List below any expired reagents, or those stored or labeled improperly.

Reagent ID _____
Reagent ID _____
Reagent ID _____
Reagent ID _____
Reagent ID _____

Comments

1. _____
2. _____
3. _____

SAC1:1/10/11: bm

11.0 Facilities, Equipment, Security, Safety and Waste Management

11.1 Facilities, Security, and Safety

CompuChem is located in Cary, North Carolina on an eleven-acre site. The laboratory facility has over 30,000 square feet of office and laboratory space (Figure 11.1). An adjacent 1,000 square foot concrete block storage building is used for waste containment. This building is vented and has a four-inch concrete berm designed to contain at least a 10% capacity spill.

Laboratory areas are separated as outlined below. A comprehensive instrument and support equipment list is found in Tables 11.1 to 11.7.

11.1.1 Sample Receiving Area

Located in the sample receiving area **are** a large walk-in refrigerator and several reach-in refrigerators for sample storage, an ambient storage unit for sample storage, and fume hooded bench space used to unpack and inventory samples received for analysis. **Freezers are also located in the area for biological tissue storage and long-term soil sample storage, if requested by the client.**

11.1.2 Sample Preparation Laboratory

The sample preparation laboratory houses both organic and inorganic sample preparation areas. Fume hoods separate the two areas. A Liebert air handling system conditions the air by heating, cooling, filtering, humidifying and de-humidifying as needed. The room is maintained under a negative pressure to isolate any solvent vapors from the analytical laboratories.

11.1.3 Semivolatile and GC Analytical Laboratory

The semivolatile/**GC** analytical laboratory houses the GC and GC/MS instrumentation used to analyze sample extracts for semivolatile, pesticide, herbicide, polychlorinated biphenyl, and diesel range organic **and RSK** compounds. A Liebert air handling system conditions the air by heating, cooling, filtering, humidifying and de-humidifying as needed.

11.1.4 High Performance Liquid Chromatography Laboratory (HPLC)

The HPLC laboratory houses the HPLC instrumentation used to analyze sample extracts for polycyclic aromatic hydrocarbons and explosives. A Comfort Maker air conditioning unit cools the space.

11.1.5 Volatile Analytical Laboratory

The volatile analytical laboratory houses the GC and GC/MS instrumentation used to analyze samples for volatile organics and gasoline range organics. A **Carrier** air handling system conditions the air by heating, cooling, filtering, humidifying and de-humidifying as needed. In an effort to eliminate cross-contamination, the **air handling system** is equipped with carbon filters and the laboratory is under positive air pressure.

11.1.6 Organic Standards Laboratory

Located in the organic standards laboratory are reach-in refrigerators and **a** freezer used for standard storage **that are segregated depending on the laboratory for which the standards are intended**. A Mettler analytical balance is used to weigh neat materials **and all standards are prepared in a fume hood**. A NIST-traceable thermometer is used for laboratory thermometer and IR gun calibrations.

11.1.7 Inorganic Analytical Laboratories

The inorganic analytical laboratories house ICP, ICP-MS, cold vapor, ion chromatograph and spectrophotometric instrumentation used to analyze sample digestates for trace metals, cyanide, minerals, nutrients, and anions. A Lennox air handling system conditions the air in the ICP lab by heating, cooling, filtering, humidifying and de-humidifying as needed.

11.1.8 Preventive and Routine Maintenance of Analytical Instrumentation and Support Equipment

The laboratory has an analytical instrument specialist on-site. He performs preventive and/or major maintenance (e.g. replacement of turbo pump oil or replacement of circuit boards) on the laboratory's analytical instrumentation. Routine instrument maintenance (e.g., replacement of injector liners and septa) **are** preformed by the analysts. Procedures for routine instrument maintenance are detailed in the SOPs for the particular analytical method and in the manufacturers instrument manuals.

Analytical support equipment such as balances, ovens, and refrigerators are monitored by the analysts using NIST-traceable reference materials. Qualified outside servicepersons calibrate each balance on-site semiannually. The NIST-traceable reference thermometer and **all** weight sets are calibrated annually by the North Carolina Department of Agriculture and Consumer Services Standards Division.

Any analytical instrument that cannot be successfully calibrated before the next work shift must be identified as out of service. The failed calibration is documented in the instrument run log. If the instrument cannot be returned to service after maintenance by the analyst, instrument specialist, or outside service person, a **tag-out** sign indicating the instrument is out of service is affixed to the instrument.

11.1.9 Documentation

The QA department issues bound and paginated log books to the laboratory areas. These log books include run logs, **equipment** maintenance **and repair** logs, temperature logs, standard preparation logs, and balance calibration logs. The analysts make entries in to these logs and return the completed logs to the QA department for archival **by the QA specialist**.

11.2 Facilities Maintenance and Security

The facility is maintained under the direction of the Manager, Purchasing, Administration, and Facilities. Preventive maintenance, repair and/or replacement of facility environmental systems such as heating and cooling units are accomplished by maintenance personnel and outside contractors under his supervision. Plans for facility renovations and improvements developed by laboratory management and staff are coordinated and implemented under his supervision.

The facility is secured by locked doors accessed by electronic and manual keys. An electronic security system monitors access. A chain link fence surrounds the entire acreage. The fence is equipped with two gates secured by pad locks during non-business hours. Only authorized personnel have unsupervised access to the facility. Security cameras have been installed at various locations on the premises and inside the laboratory.

11.3 Safety

Laboratory personnel **are required to** wear protective eyewear, lab coats, and gloves while handling samples. Emergency showers are located in the sample preparation laboratory. Eye wash stations and fire extinguishers are located throughout the facility. The safety committee performs monthly safety inspections and reports observations to management. A list of safety equipment is presented in **Table** 11.8.

11.4 Contingency

Progress Energy Carolinas, Inc. supplies electrical service to CompuChem. The laboratory also maintains an uninterruptible power supply (UPS), that powers computers, instruments, and hoods for approximately two hours in case of a temporary power outage. The laboratory has at its disposal a refrigerated tractor trailer for maintaining samples at the required storage temperature. Dry ice would be used to maintain freezer temperatures.

Qualified subcontracting laboratories are available to perform analyses on short notice in emergencies. In such a case, CompuChem notifies clients and gains approval before allowing a subcontractor laboratory to perform analyses.

11.5 Reagent Storage Control, Documentation and Labeling

The receipt date, open date, expiration date, and the initials of the person recording the information are placed on reagent bottle labels after receipt by the laboratory. If the vendor does not supply an expiration date, a period of **five** years from date of receipt is used. Table 11-9 summarizes the reagents present at the laboratory and under what conditions they are stored. Before a solvent lot is purchased, it is analyzed and approved by the laboratory. Once the lot is approved, the vendor supplies the laboratory with reagents until that lot is depleted.

In methods where the purity of reagents is not specified, **American Chemical Society (ACS)** grade is used. Reagents of lesser purity than those specified by the method are not used. Reagent container labels are checked to verify that the purity meets the requirements of the particular method. Reagent lot numbers are documented on preparation sheets and instrument run logs **and through the LIMS**.

11.6 Hazardous Waste

Waste handling and disposal procedures are carried out in compliance with CompuChem's waste disposal SOP (SOP 12.1: "Hazardous Waste Disposal"). This SOP includes procedures for training of personnel, identification, segregation, storage, and details of disposal procedures. Disposal of samples, sample digestates, and sample extracts is detailed in sample control SOPs. Waste disposal is performed in compliance with all applicable local, state, and federal regulations. A licensed hazardous waste contractor handles all recycling and arrangements for final disposal. (See Table 11-10.)

CompuChem is subject to yearly RCRA inspections by the State of North Carolina Hazardous Waste Section. CompuChem is a large quantity waste generator (in excess of 1000 kg per month) and must comply with all applicable parts of the following regulations:

- Resource Conservation and Recovery Act (RCRA)(40 CFR 261-271)
- Occupational Safety and Health Administration (OSHA)(29 CFR 1910.120 and 1910.1200)
- Hazardous Material Transportation Act (HMTA)(49 CFR 171-180: HM-181 and HM126F)
- Clean Water Act (CWA)(40 CFR 403.5)
- Superfund Amendments Reauthorization Act (SARA)(40 CFR 355, 370)
- NC Hazardous Waste Rules(15A NCAC 13A)

CompuChem pursues waste minimization efforts and makes every effort to recycle the materials and chemicals that are well suited for this purpose. After analysis and following a 90 day holding period, all residual solid and aqueous samples are disposed of as hazardous waste. Wastes are segregated into various waste streams which fall under RCRA codes F001, F002, F003, F005, D001, and D018. Table 11.10 represents general waste disposal procedures.

Any additional wastes not listed above are evaluated by the hazardous waste technician, safety officer, and/or contracted disposal company to determine the appropriate means of disposal.

Each satellite accumulation container must have a hazardous waste label. Shipment containers must be labeled according to HM-181 and must include the generator's name and address, EPA ID number, manifest document number, accumulation start date, and EPA waste number. All hazardous waste shipments are accompanied by a North Carolina Hazardous Waste Manifest. One copy of the manifest is kept on file at CompuChem at the time of shipment by the hazardous waste technician. Additional copies accompany the waste to the hazardous waste contractor. After disposing of the waste, the contractor returns a signed copy of the manifest to CompuChem verifying disposal. These final copies of the manifests are kept on file by the hazardous waste technician. Logbooks are maintained for daily inspections of the outside 90-day waste storage building and manifest tracking and are located inside the waste storage area. To prevent any improper disposal, the licensed hazardous waste contractors perform additional testing on the contents of the shipment containers before disposal.

Figure 11-1
Laboratory Floor Plan

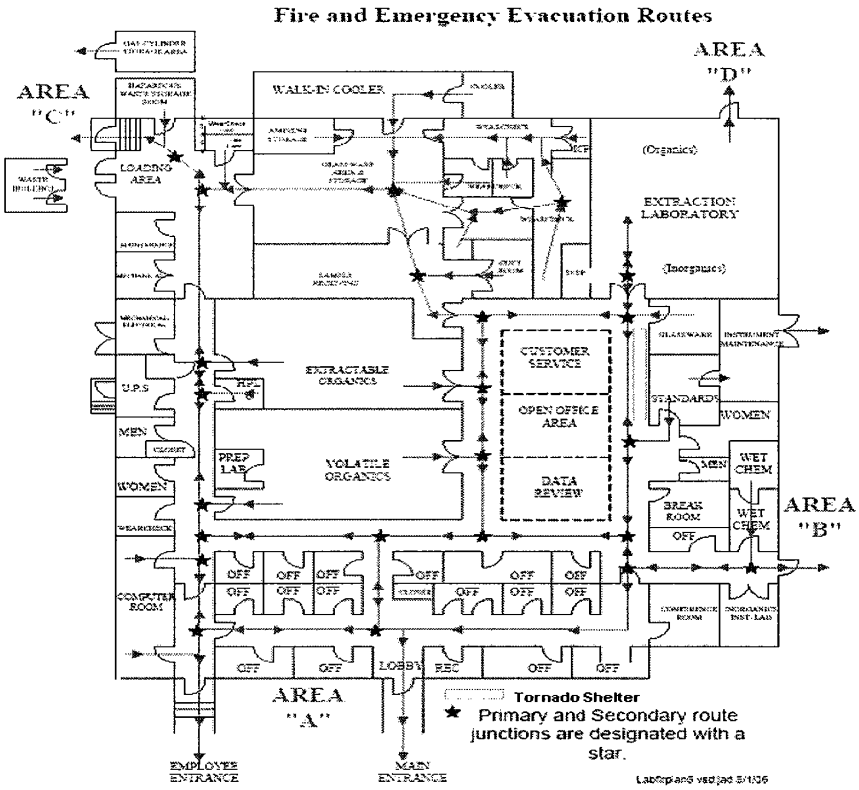


Table 11.1 GC Laboratory Equipment

Model No.	Serial No./Channel No.	Type	Installation Date
GC			
Varian 3400	#54	NPD	
Varian 3400	6949 #37/42	Dual ECD	1989
Varian 3400	3623 #39/41	Dual ECD	1987
Varian 3400	17403 #44	FID	
Varian 3400	3356 #59	FID	
Trace GC 20011612	#86/87	Dual ECD	2000
Trace GC	TR101370/#80/81	Dual ECD	2000
Trace GC	TR101369/#82/83	Dual ECD	2000
Trace GC	TR101375/#84/85	Dual ECD	2000
Hewlett Packard P5890	Series II GC LR47359C #67	FID	2002
Hewlett Packard 5890	Series II GC #32/33	Dual ECD	
Agilent Technologies 6890N	CN10552045 #90/91	Dual ECD	2006
Agilent Technologies 6890N	CN10522046 #92/93	Dual ECD	2006
Autosampler			
CTC A2000S	1304/89(#37/42)	Autosampler	1991
CTC A2000S	13013(#39/41)	Autosampler	1991
CTC A2000S	1301/89(#44)	Autosampler	1991
CTC A2000S	12371(#54)	Autosampler	1989
CTC A2000S	1792/90(#59)	Autosampler	
CTC A2000S	1915/90(#86/87)	Autosampler	1991
CTC A2000S	12369(#80/81)	Autosampler	2000
CTC A2000S	1804/90(#82/83)	Autosampler	1990
CTC A2000S	12387(#84/85)	Autosampler	2000
CTC HS500	30135(#67)	Autosampler	2002
CTC A2000S	12371 (#32/33)	Autosampler	
Agilent Technologies 7683B	CN54637426 #90/91	Autosampler	2006
Agilent Technologies 7683B	CN62940489 #92/93	Autosampler	2006
Refrigerators			
Refrigerator #2	GC Lab Refrigerator #2	8 ft^3	
Refrigerator #7	GC Lab Refrigerator #7	8 ft^3	
Refrigerator #8	GC Lab Refrigerator #8	22,7 ft^3	
Refrigerator #3	GC Lab Refrigerator #3	8 ft^3	
Refrigerator #4	GC Lab Refrigerator #4	22 ft^3	
Refrigerator #9	GC Lab Refrigerator #9	22 ft^3	

Table 11.2 HPLC Laboratory Equipment

Model No.	Serial No./Channel No.	Type	Installation Date
Agilent 1100 Series HPLC Micro Vacuum Degasser Quaternary Pump ALS Sampling System Thermo Column Compartment Multiple Wavelength Detector	a11001 JP40714429 DE40926792 DE33224733 DE405384472 JP43826437	HPLC	2004
Agilent 1100 Series HPLC Micro Vacuum Degasser Quaternary Pump ALS Sampling System Thermo Column Compartment Multiple Wavelength Detector	a11002 JP40721307 DE43631227 DE43627396 DE43644464 JP43826438	HPLC	2005
Refrigerators			
Baxter Explosion-Proof		Precision 814, 8 ft^3	
Marvel #1		8 ft^3	

Table 11.3 SV GC/MS Laboratory Equipment

Model No.	Serial No./ID No.	Type	Installation Date
Hewlett Packard HP5972 GCMS 5890 Series II GC HP 7673 autosampler	#60 3244A32866	SV	1994
Hewlett Packard HP5972 GCMS 5890 Series II GC HP 7673 autosampler	#66 3442A40444	SV	1996
Hewlett Packard HP5972 GCMS 5890 Series II GC HP 6890 autosampler	#70 3529A43269	SV	1998
Hewlett Packard HP5972 MSD 5890 Series II GC 6890 Series injector	#62 3404A37674	SV	2005
Hewlett Packard **HP5972 GCMS** **5890 Series II GC** **6890 Series injector**	**#64** **3415A38536**	**SV**	**GCMS 1996 in SV as #64; moved to VOA in 2008 as #69 Returned to SVOA as #64 in 2010**
Agilent Technologies 6890N Network GC System (G1530N) Agilent 5973 Network Mass Selective Detector (G1530N) **Agilent Autosampler (7683B)**	#68 US10143053 CN 83150389	SV	2002 in VOA as #61/62; moved to SVOA in 2008 as #68
Refrigerators			
Refrigerator #1	GC/MS SVOA Lab Ref. #1	8 ft^3	
Refrigerator #3	GC/MS SVOA Lab Ref. #3	22 ft^3	

Table 11.4 VOA GC/MS Laboratory Equipment

Model No.	Serial No./ID No.	Type	Installation Date
Finnigan INCOS 500 GCMS Tekmar 3000 concentrator Archon autosampler	#55 IN001125 95107004	VOA	1991
Hewlett Packard HP6890 MSD 6890 Series GC Tekmar 3000 P&T concentrator Varian Archon autosampler	#71 US00004710 95097006 610701	VOA	2000
Hewlett Packard HP5972 MSD 5890 Series II GC Varian Archon autosampler, closed system P&T Tekmar LCS 3000	#59	VOA	2000
Hewlett Packard HP5973 MSD Varian Archon autosampler Tekmar/Dohrmann 3100 sample concentrator	#90	VOA	2005
Hewlett Packard HP5972 MSD 5890 Series II Plus GC Tekmar 3000 P&T concentrator Varian Archon autosampler	#73 700758 94293001 507204	VOA	1/2001
Agilent Technologies Inert 5975 MSD 6890N Network GC System	#91 US54431647 CN10604034	63172A G1530N	2006
Varian 3400 Tekmar LCS2000 Tekmar ALS2016	6952 (#68) 90163020	FID Purge & Trap	1989
Varian 3400 Tekmar LCS2000 Tekmar ALS2016	6954 (#58) 90038029 91158005	FID Purge & Trap	1990

Table 11.4 VOA GC/MS Laboratory Equipment (continued)

Model No.	Serial No./ID No.	Type	Installation Date
Refrigerators			
Refrigerator #2B			
Refrigerator #4/Freezer #3		22 ft^3	
Refrigerator #5	880101750		
Refrigerator #6	9910245654	5 ft^3	
Freezer #6		22 ft^3	
Water System			
Millipore Milli Q	06731-C		
Oven			
Blue M			
Weights			
Troemner	45823	Class 1	
Balance			
Sartorius B410	40010010	Electronic	

Table 11.5 Inorganics Laboratory Equipment

Model No.	Serial No./ID No.	Type	Installation Date
TJA Trace ICAP P3 Trace ICAP RF Generator Chiller Autosampler ThermoSPEC software	303490 Revision 6.20	**61E Trace Vacuum** Simultaneous	1994
TJA Trace ICAP P4 36161 System **RF Generator** **Chiller** **Autosampler** **ThermoSPEC software**	10670/13685200 **Revision 6.20**	61E Trace purge, **Simultaneous**	2000
Perkin Elmer Sciex Elan **DRC-e PEMS** Elan Software Polyscience Recirculator **Elemental** Autosampler	AH13950808 **Version 3.4**	ICP-MS **DRC-e**	2008
Mercury Analyzer Leeman Labs **V4** Hydra AA **WinHg Software**	7031 **Version 1.7**	CVAA Automated Mercury Analysis System	2007
Buck Scientific Oil in Water IR Analyzer	506	**IR Analyzer**	**1994**
Dionex Ion Chromatograph **IC2** AS40 Automated Sampler **Chromeleon Software**	U503481/IC2 **Version 1.2**	**Ion Chromatograph**	2005

Table 11.5 Inorganics Laboratory Equipment (continued)

Model No.	Serial No./ID No.	Type	Installation Date
TOC Analyzer			
Shimadzu TOC-V	H51104335168	VCSH	2006
Shimadzu Solid Sample Module	H52504300074	SM-5000A	
Shimadzu Autosampler	H52104301662	ASI-V	
Client Pro PC	4169020-001		
Lachat Instruments	A5000-415	Ion Chromatograph	
IC A5000			
CETAC ASX-510 Auto Sampler	100124 ASX		2009
Quickchem	2000-0076	2300-000	1990
Proportioning Pump	2000/12	2200-00	
PC Hewlett Packard	3024A01573	Vectra 286/12	
Printer Hewlett Packard	2935A70310	LaserJet Series II	
Zellweger Analytics, Inc.			
QuickChem FIA+			
8000 series	A83000-1605		2000
Spectrophotometer			
Spectronic 21D	3152073028	Spectrophometer	
Milton Roy			
Balances			
Sartorius	10505094	LC2200P/Electronic	1993
American Scientific	2904542	S/P 180/Electronic	1993
O'Haus E400	2061	Electronic	1993
O'Haus E400I	6371	Electronic	1993
Demoer		Electronic 4 place	
Mettler	H50747	PJ300	
Denver Instruments	B034903	300	
Weights			
Troemner	NCSN050	Class 1	
Fisher	NCSN054	Class 1	
Wet Chemistry Lab Refrigerator #1		8 ft^3	
Ovens			
Fisher ISO Temp200 Series	00143		
Fisher ISO Temp200 Series	00179		
Miscellaneous			
Electrothermal		10 place midi	
Midivap system (2)		distillation	
Distillation apparatus (6 units)			

Table 11.5 Inorganics Laboratory Equipment (continued)

Model No.	Serial No./ ID No.	Type	Installation Date
Miscellaneous (continued)			
Hot Plates (seven units)			
Fisher pH Meter	50/PH1	Accumet	
Block Digestors			
Lachat	BD46		
Environmental Express SC100 w/SC200 racks		36 well block digestor	10/97
Baths			
Digestion (2) turkey roasters Fisher Versa Bath, Precision Stainless		183	
Pyrex Burets		10, 25, 50 mL	1999
Ovens			
Fisher Isotemp Oven	00700173	Mode 500 Series	
Fisher Isotemp Oven	472	Mode 200 Series	
Fisher Scientific Dessicator Unit		**12" x 12" x 10 3/4"**	**2010**
Flash Point Instrument Koehler Instruments, Inc.	R02291814	K16200	2006
Electrothermal Electromantles (4 units, 15 place)		ME	
Fisher Centrifuge	00200499	Centrific 228	
Corrosivity testing apparatus		Glass vessel	
Refrigerators			
American Scientific Products	NY 79522-2	SPR 24 A-O-A	
Gerald	6081295V	GR 17A	
Microwave Digestion			
Acid Distillation Unit			

Table 11.6 Organic Sample Preparation Laboratory

Model No.	Serial No./ ID No.	Type	Installation Date
Balances			
O'Haus E400	3156	Electronic	
Sartorius BL310	13008591	Electronic	3/2000
Weights			
Troemner	45824	Class 1	
pH Meters			
pH Meter - Beckman	2721	Hand held	
pH Meter – WTW	3204	Hand held	
Baths			
Branson ultrasonic bath	B-22-4		
Branson ultrasonic bath 8510	RPA 9806516E		
Hot water baths (2 units/cp. 24)			
Ovens			
Fisher Econotemp	477	55G	
Wilt Furnace	90870	210	
Refrigerators			
Whirlpool	EE02722440	Compact	
Centrifuges			
IEC	24760668	Centra 8	
IEC	24760672	Centra 8	
Fisher Centrifuge	1041	225	
Water Purification System			
US Filter DI Water System (250 gal)			
Other			
Fisher Dessicator			
YSI Conductivity Meter	1422	Model 35	
Sonifiers			
Branson Sonifier	NH10245B	350 Four Tips	
Branson Sonifier	NH20102B	350 Four Tips	
Tekmar	11896E2	Model TM 600-2	
Tekmar	10500	Model VC 600-2	
Tekmar	13771B	Model TM375	
Nitrogen Evaporators			
Organomation	8373	115	
Separatory Funnel Shaker			
Glas-Col® Funnel Holders	**471150,-51,-52, -53/Cat. # 099A VH2000DS**	**Capacity for Eight 2 L Separatory Funnels**	**2010**
Glas-Col® Floor Shaker Base	**471162/Cat. # 099A VS5504**	**Variable Speed, 3D Motion**	**2010**

Table 11.6 Organic Sample Preparation Laboratory (continued)

Model No.	Serial No./ ID No.	Type	Installation Date
Environmental Express Extraction Vessels and rotation units			
ZHE			
3 rotation units/8 vessels each			
TCLP/EP Toxicity			
3 rotation units/8 vessels each			
Gel Permeation Chromatograph			
ABC GPC #1	834B-234	1002B	1990
ABC GPC #2	879B-279	1002B	1991
GPC #6	GPC-6	J2 Scientific	2007
Autosampler Model # J2AIM3310	4738A12420		
AccuPrep MPS GPC Unit	07J-1273-4.1DI		
GPC #5	GPC-5		
Autosampler Model # J2AIM3310	4738A12422	J2 Scientific	2007
AccuPrep MPS GPC Unit	07K-1275-4.1DI		
Recirculating Chillers			
Neslab	8005036	Cool flow 75	
Neslab		Cool flow CFT-75	
Neslab		HX-75	
Automated Soxhlet			
Gerhardt Soxtherm	4032297/4032044	SE-416	
Gerhardt Soxtherm	4022028/4022069	SE-3A/S306A	2003

Table 11.7 Organic Standards Laboratory Equipment

Model No.	Serial No./ ID No.	Type	Installation Date
Weights			
Permas	A302	Class 1	
Balance			
Mettler H31AR	794794	Electronic Analytical	

Table 11.8 Safety Equipment

Model No.	Serial No./ ID No.	Type	Availability
Fire Extinguishers	Various	General Fire Ext., Inc.	**Throughout the facility**
Eye Protection			
Safety glasses		Safety glasses	One per person plus surplus
Eye-wash stations		Model 3729	**Six** portable; two permanent
Protective Clothing		Lab coats North Safety Equipment	One per person, plus surplus
Respirators			Two
Safety Showers		Water	Two in the sample prep lab

Table 11.9 Reagent Storage

Reagent	Storage	Location	Conditions
Halogenated solvents	Vented storage cabinets	Inorganics Extractions Extractable GC/GCMS Standards Electronics	Air conditioning
Non-halogenated solvents	Vented storage cabinets	Inorganics Extractions Extractable GC/GCMS Standards Lab Electronics	Air conditioning
Alcohols	Vented storage cabinets	Inorganics Extractions Volatile Preparations Extractable GC/GCMS Standards Electronics	Air conditioning
Inorganic chemicals	Shelving cabinets	Inorganics Extractable GC/GCMS Standards Electronics	Air conditioning
Trace metals	Shelving cabinets	Inorganics	Air conditioning
Peroxides (H_2O_2)	Vented storage cabinets	Inorganics	Air conditioning
Acids (Inorganic)	Vented storage cabinets	Inorganics	Air conditioning
Acids (Organic)	Vented storage cabinets	Extractions Inorganics	Air conditioning
Organic standards	Explosion-proof refrigerators	Volatile GC/GCMS Extractable GC/GCMS Standards	Air conditioning
Stock organic solvents	Vented storage cabinet	Extractions	Air conditioned
Stock inorganic chemicals (NaOH)	Vented storage room	Solvent storage building	Vented ambient outside storage
Stock acids	Vented storage room	Solvent storage building	Vented ambient outside storage

Table 11.10 Waste Disposal

Waste Stream	Storage	Disposal
Dichloromethane	55-gallon steel drums	Reclaimed
Freon 113	55-gallon steel drums	Reclaimed
Used oil	55-gallon steel drums	Reclaimed
Mixed flammable solvents	55-gallon steel drums	Disposed by HW contractor
Vials with flammable solvents	55-gallon steel drums	Disposed by HW contractor
Plant scraps with solvents	55-gallon steel drums	Disposed by HW contractor
019 waste water	55-gallon polyethylene drums	Disposed by HW contractor
Purged soil	55-gallon polyethylene drums	Disposed by HW contractor
Acid water and vials	55-gallon polyethylene drums	Disposed by HW contractor
Mixed acids	55-gallon polyethylene drums	Disposed by HW contractor
NaOH waste	55-gallon polyethylene drums	Disposed by HW contractor
Oil and acid	55-gallon polyethylene drums	Disposed by HW contractor
Cyanide standards	55-gallon polyethylene drums	Disposed by HW contractor
Pyridine and barbituric acid	55-gallon polyethylene drums	Disposed by HW contractor
Metal standards	55-gallon polyethylene drums	Disposed by HW contractor

12.0 Corrective Action

Corrective action is the process of identifying the root cause and implementing the procedures to correct nonconforming work and departures from the policies and procedures of the laboratory's quality system. The need for corrective action may arise from the identification of any of the following:

- Nonconformance issues identified during sample processing and analysis
- Deficiencies in the laboratory's quality system as identified as a result of internal and external audits
- Failure to achieve acceptable proficiency testing (PT) results
- Client inquires and complaints

Corrective actions are documented in Corrective Action Reports (CARs) (Figure 12-1), memoranda, management reports (including responses to external audit reports), instruments logs, and work-order/SDG narratives.

If issues arise that potentially compromise the integrity of the validity of test results, the issues are immediately brought to the attention of either the QA Manager or the Vice President for resolution. If an issue cannot be resolved internally and/or satisfactorily, the client is contacted within 72 hours of the identification of the event.

12.1 Nonconformance Issues Identified During Sample Processing and Analysis

12.1.1 Sample Receipt

Nonconformance issues identified during sample receipt may include but are not limited to the following:

- Samples received at temperatures outside of acceptable range
- Samples received improperly preserved
- Samples received in damaged or broken containers

In cases such as those listed above, receiving personnel document the nonconformance on the client chain-of-custody (COC) and advise the **appropriate** project manager of the problem. The project manager then immediately contacts the client. The project manager documents the client's instructions in writing on the client COC or separate document. **See Section 5.0 for further details on sample receipt and handling.**

When the lab receives a sample from a site in North Carolina, which does not meet the requirements for shipping temperature, preservation, volume, or holding time, the lab will inform the client. If the client chooses not to resample but will accept the qualified results from the laboratory, the North Carolina Department of Environment and Natural Resources Division of Water Quality must be notified. This written notification must include the sampling site, client name, client address, and the non-compliance issue.

12.1.2 Sample Preparation

Nonconformance issues identified during sample preparation may include, but are not limited, to the following:

- Insufficient sample volume for the desired test method
- Sample matrix not amenable to the desired test method
- Laboratory accident during sample handling resulting in loss of sample extract/digestate
- Improper addition of QC reference standards during sample preparation

In cases such as those listed above, the sample preparation technician advises the project manager of the problem and the procedure continues as stated above.

The technician documents instances such as an alternate sample volume used for preparation, a laboratory accident, or the addition of the incorrect volume of QC reference standard on the preparation worksheet.

12.1.3 Sample analysis

Nonconformance issues identified during sample analysis may include but are not limited to the following:

- Initial or continuing calibration fails acceptance criteria
- Field samples fail QC acceptance criteria
- QC samples fail acceptance criteria
- Sample preparation or analysis holding time exceeded

In the case of the failing initial or continuing calibration, the analyst evaluates the results and may perform instrument maintenance before recalibration. This is documented in the appropriate instrument log book. Failed QC acceptance criteria for field and QC samples are documented in the instrument run log. Corrective actions for QC sample failure are detailed in the appropriate analytical SOP.

All corrective actions performed during the processing and analyses of samples are documented in the final report to the client. The work-order/SDG narrative summarizes the nonconformance issues and corrective actions taken.

12.2 Deficiencies in the Laboratory's Quality System Identified as a Result of Internal and External Audits or Proficiency Testing Results

The laboratory's quality system is monitored through the periodic performance of internal and external audits and proficiency testing (PT) samples. Deficiencies in the laboratory's quality system affecting its ability to produce data of known and documented quality may be identified as a result these audits.

Deficiencies identified as a result of internal and external audits may include, but are limited to, the following:

- Deviations from established laboratory policies and procedures
- Deviations from the requirements of the NELAC standards or other external agency requirements
- Deviations from the requirements of the appropriate CLP statement of work

Deficiencies identified during internal audits are documented in internal CARs, interoffice memoranda, internal audit reports, and monthly management reports.

Deficiencies identified during external audits, both on-site and through data and electronic media audits, are documented in external audit reports prepared by the auditing body and internal CARs.

For both internal and external audits and PT deficiencies, QA **Department** issues an internal CAR to the responsible manager/supervisor. The responsible manager/supervisor then identifies the root cause of the deficiency and **initiates** a plan of correction. The **root cause and** plan of correction **are** documented on the CAR **report form and** is returned to the QA department for approval. For an external audit, the laboratory's plan of correction for deficiencies is documented in a written response to the auditing body for its acceptance. Additionally, corrective action reports for failed PT results are submitted to the appropriate accreditation bodies. All records are **tracked and** retained.

12.3 Client Inquiries or Complaints

CompuChem's clients are represented at the laboratory by Project Managers. The Project Manager **or Vice President of Sales and Marketing are** the client's point of contact for any questions and/or complaints.

For issues that arise between the time that the client samples are received by the laboratory and the time the final report is completed, the client contacts the Project Manager with any questions or changes. The Project Manager relays the information to the responsible manager/supervisor. This client correspondence is documented in the Project Manager's **message notebooks and e-mail communications**.

For issues that arise after the final report has been delivered, the client again contacts the Project Manager. The issue is **initiated via the** Client Inquiry **system** that is forwarded to the responsible supervisor/manager for resolution.

Issues cited by the client may include but are not limited to the following:

- Missing or illegible page(s) in the final report
- Errors in sample processing and analysis
- Request for further analysis on sample previously analyzed
- Request for further information such as a more detailed report style than originally requested and electronic disk deliverable (EDD)
- Request an additional copy of the report

Once the issue has been **addressed** and the resolution documented**,** any supporting documents are returned to the Project Manager **or designee**. The Project Manager forwards the information to the client. If an amendment of the final report is required, it is identified as such before being submitted to the client.

12.4 Permitting Departures from Laboratory Policies and Procedures

12.4.1 Sample Deviations

The Vice President and General Manager and the Manager of QA write laboratory policies as the need arises. There are instances, during the course of normal operations, when the laboratory may be allowed to deviate from routine standard operating procedures, internal laboratory policies, or client

specifications. An example of this would be if an analyte fails to meet QC limits in the LCS and is of **no** interest to the client for **a particular** project. In these instances, the laboratory staff must speak directly with, and obtain permission from, the Vice President and General Manager or the Manager of QA and the client, if there is questionable impact on the data. Laboratory staff **are expected to consult the** QA **Department** to seek clarification and direction on situations that occur outside normal **operating** procedures. Project Managers maintain regular communication with clients regarding status of projects, sample analysis, **and report criteria**.

A laboratory supervisor or analyst who has a question or **concern** about a **procedural or protocol** deviation is appropriate contact**s the** QA **department** to discuss resolution of the matter. The QA staff member will evaluate the situation relative to documented policies and procedures to determine if the data may be reported, or **if** corrective action must occur. A recommendation is made regarding the course of action including contacting the client, **if necessary**. In general, any allowed deviation is discussed in the SDG narrative **of the client's final report**.

12.4.2 Logbook Deviations

The following statement has been added to all logbooks, "The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation."

Please Note: This section does not apply to samples analyzed for the Ohio Voluntary Action Program (Ohio VAP). Deviations from Ohio VAP approved laboratory SOPs are not permitted. The Ohio VAP must approve any proposed revisions to previously approved SOPs prior to implementing any changes in the laboratory.

12.5 Preventative Action Report

Preventative action is the process of identifying root causes and implementing the procedures to identify and address potential nonconforming work and departure from the policies and procedures of the laboratory's quality system before corrective action has to be taken. The identification of a preventative action is similar to that of a CAR. Preventative actions are documented in Preventative Action Reports (PARs) (Figure 12-2).

Figure 12-1

CORRECTIVE ACTION REPORT

CompuChem
A Division Of
Liberty Analytical Corp.

CORRECTIVE ACTION REPORT

INITIATION

Project:	Date Initiated:
Client Name:	Required Response Date:
Initiator:	CAR#
Deficiency Type: (circle one) CERT – GLP – MDL – NARR – PT – RPT – REV – SOP – SOW – QC	
Recipient:	Lab Area:

NON-CONFORMANCE

Description of Non-Conformance:

ROOT CAUSE

Signature of Responsible Supervisor/Manager:	Date:
Reason for Non-conformance:	

CORRECTIVE ACTION

Signature of Responsible Supervisor/Manager:	Date:
Plan of Correction (Must Address the Root Cause):	
Target Implementation Date:	

IMPLEMENTATION OF CORRECTIVE ACTION

Auditor:
Does SOP need revision? If so, target date of draft revision?
CAR Response Date:
CAR Closure Date:

FOLLOW-UP (completed by QA)

Auditor:	**Audit Date:**
Follow-up findings:	

Car4 –12/07/09: jr

Figure 12-2

PREVENTATIVE ACTION REPORT

CompuChem
A Division Of
Liberty Analytical Corp.

PREVENTATIVE ACTION REPORT

INITIATION

Initiator:	Date Initiated:
Recipient:	
Lab Area:	PAR#
Deficiency Type: (circle one) CERT – GLP – MDL – NARR – PT – RPT – REV – SOP – SOW – QC	

POTENTIAL NON-CONFORMANCE

Description of Potential Non-Conformance:

ROOT CAUSE

Signature of Responsible Supervisor/Manager:
Reason for Potential Non-conformance:

PREVENTATIVE ACTION

Signature of Responsible Supervisor/Manager:	Date:
Plan of Action (Must Address the Root Cause):	
Target Implementation Date:	

IMPLEMENTATION OF PREVENTATIVE ACTION

Auditor:
Does SOP need revision? If so, target date of draft revision?
PAR Submittal Date:
PAR Closure Date:

FOLLOW-UP (completed by QA)

Auditor:	**Audit Date:**
Follow-up findings:	

PAR2 –6/25/10: jr

13.0 Subcontracting and Support Services and Supplies

CompuChem only uses those outside support services and supplies that are of **satisfactory** quality to sustain confidence in the laboratory's tests. Records of all suppliers, for support services or supplies required for tests, are maintained.

13.1 Control of Subcontracted Analytical Services

CompuChem performs a wide array of analytical methodologies, and **occasionally** the laboratory must locate a suitable alternate laboratory to perform methods not performed in house. Only NELAP accredited laboratories are used to perform subcontracted analyses. In addition to NELAP accreditation, subcontract laboratories utilized by the lab must be assessed and approved by the Department of Defense **(DoD)** component for **DoD projects**. CompuChem's client agrees to the use of a qualified laboratory for subcontracted analytical services before the samples are sent off-site. The Project Management team coordinates subcontract laboratories on a project-by-project basis through agreement and approval of our client. An evaluation of the laboratory may include a review of standard operating procedures, Quality Manual, Statement of Qualifications, and recent PT study scores. The **contracted** lab's performance may be monitored through data evaluation.

13.2 Material Procurement and Control

Two prime objectives **for** CompuChem's purchasing are maintain**ing** sufficient **levels of** supplies **and evaluating** the best total value in a combination of supply, price, required quality, and service. Department and laboratory supervisors have primary responsibility for maintaining adequate inventory of supplies and ensuring that all supplies/equipment meet or exceed quality requirements. Supervisors work through the purchasing agent to meet these objectives. **Records of all suppliers for support services or supplies required for tests are maintained.**

CompuChem uses competitive inquiries or requests for bids, along with appropriate negotiation, to provide equal opportunities for potential and current suppliers to earn CompuChem's business and to allow the laboratory to seek the best overall value. Long-term considerations include reliability, price, required quality, and service. Suppliers must maintain the confidentiality of competitively sensitive information that is obtained from the Accounting department or other CompuChem personnel. Prices and related information, whether accepted or not, are not disclosed.

13.3 Material Quality Inspection, Outside Support Services and Supplies

Laboratory personnel consult the QA department when purchasing supplies/equipment that could potentially affect data quality, and therefore results of sample analyses, before use in production. The Vice President and General Manager or Manager of QA determines the appropriate test procedures and evaluates the resulting test data. A similar validation process is used in testing new instrumentation. When variability is exhibited in the quality of vendor-supplied materials or services, the laboratory **manager or** department supervisor is responsible for working with Purchasing **in** find**ing** a suitable alternative source.

Information on new chemicals must be supplied to the chemical hygiene officer, safety officer, **and**/or the waste management officer. Items and services have been identified that are known to affect quality. Documentation of material quality inspections for solvent lot tests, is maintained in the QA department. Laboratory supervisors maintain other documentation.

Material Safety Data Sheets (MSDS) are maintained by the QA department and are required reading by staff members using a particular chemical substance.

14.0 References

14.1 2003 NELAC Standard**, July 1, 2003**

14.2 ASTM D-5283-92. Generation of Environmental Data Related to Waste Management Activities: Quality assurance and Quality Control Planning and Implementation

14.3 EPA 2185 – Good Automated Laboratory Practices, 1995

14.4 ISO 17025: 2005. General requirements for the competence of testing and calibration laboratories

14.5 Guidance for Developing Quality Systems for Environmental Programs (QA/G-1), EPA/240/R-02/008, November 2002

14.6 Guidance on Assessing Quality Systems (QA/G-3), EPA/240/R-03/002, March 2003

14.7 QA/R-5: EPA Requirements for Quality Assurance Project Plans EPA/240/B-01/003, March 2001

14.8 QA/G-5: Guidance for Quality Assurance Project Plans EPA/240/R-02/009, December 2002

14.9 U.S. EPA Contract Laboratory Program Statements of Work SOM01.2 and ILM05.4

14.10 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update III, 12/96

14.11 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Update IV, 2/07

14.12 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **10/25/10**

Appendix A

Demonstration of Capability

A demonstration of capability is made prior to using any test method, and at any time there is a significant change in instrument type, personnel or test method.

All demonstrations are documented through the use of the Demonstration of Capability Certification Statement in this Appendix.

The following steps are performed.

a) A quality control sample is obtained from an outside source. If not available, the QC sample is prepared by the laboratory using stock standards that are prepared independently from those used in instrument calibration.

b) The analyte(s) are diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified, or if unspecified, to a concentration approximately 1-4 times the limit of quantitation.

c) Four aliquots are prepared and analyzed according to the test method either concurrently or over a period of days.

d) Using all of the results, the mean recovery and standard deviation are compared to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if there are not established mandatory criteria). If all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

f) When one or more of the tested parameters fail at least one of the acceptance **criterion**, the laboratory repeats the test for all parameters that failed to meet criteria. If repeated failure occurs, the laboratory will locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

Certification Statement:

The following certification statement is used to document the completion of each demonstration of capability. A copy of the certification statement is retained in the training files of each affected employee.

CompuChem
A Division Of
Liberty Analytical Corp.

Demonstration of Capability
Certification Statement

Study Date:
Laboratory Name: CompuChem, a Division of Liberty Analytical Corporation
Laboratory Address: 501 Madison Avenue, Cary, NC 27513
Analyst Name/Employee ID#:

Matrix:
Method Number:
SOP No./Rev#:
Analyte/Class of Analytes/Measured Parameters:

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method, which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method was performed by the analyst identified on this certification.

3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration capability are true, accurate, complete and self-explanatory.

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Technical Director's Name and Title	Signature	Date
Quality Assurance Officer's Name	Signature	Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing.
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Name:Date
Analysis: DOC
Prep:DOC

DOC1 02/25/10:jr

Appendix B

Procedure for Demonstration of Capability

CompuChem's Initial and On-going Demonstrations of Capability (Analyst Qualification) Policy

This policy details procedures for documenting the initial demonstration of capability (IDOC) and the on-going demonstration of capability (ODOC) for analysts in the laboratory following the rules listed below.

Initial Demonstration of Capability

General

1) Trainees are not allowed to process samples independently before they have demonstrated capability to perform the method through acceptable precision and accuracy testing with adequate documentation.

2) During the on-the-job training period no trainee is allowed to sign for his or her work without the signature of a manager/supervisor or an approved analyst, i.e., one who has documented capability for the method and has received approval to process samples independently. The approved analyst must cosign with the trainee on the instrument run logs or extraction worksheets.

3) The manager/supervisor, or designee, will provide the IDOC documentation to the QA department. Documentation includes all associated raw data, log sheets, and precision and accuracy statistics.

4) The precision and accuracy statistics must include the replicate values **expressed in concentration, standard deviation as % relative standard deviation (RSD) of the concentrations, the true value, and a mean percent recovery from the true value**.

5) The acceptance criteria for precision and accuracy are those stated in the methods or in CompuChem's Standard Operating Procedures (SOP) and Quality Manual. Percent recovery criteria for the LCS and RSD values for **second source** initial calibration **verification (ICV)** standards will be applied to **ICV standards for instrument analysts** where the extracted LCS recovery may be inappropriate. The Initial Calibration Verification (ICV) criteria are appropriate in this application.

   If any failure occurs, the error must be corrected and the study repeated for the failed analyte(s). This may involve only a reanalysis or a re-preparation followed by analysis. Approval is based on an individual analyte basis.

6) Approval must be made by the QA Department and documented through the NELAC Demonstration of Capability Certification Statement. This form must be signed by both the supervisor and QA Department and is maintained in the employee's training files.

7) Failure to have an IDOC on file for an employee who processes samples **may** result in rejected data.

8 **Major revisions to methods will require new IDOCs.**

9 **Department of Defense (DoD) criteria must be met to satisfy the IDOC requirement for DoD methods.**

10 **The successful performance of a Limit of Quantitation Study (LOQ) can be used to satisfy the IDOC requirement.**

11 **Work orders are required to be generated for all IDOCs performed in order that the appropriate data package is generated.**

11

GC/MS and GC Volatiles

1) For GC and GC/MS volatiles, **four consecutive continuing standards may be used to compile the IDOC, if the LCS is prepared from second source standards. Four analyses of a second source ICV may also be used to compile the IDOC.**

2) For GC/MS volatile, analysis of an IDOC by any current method will give the analyst credit for all **remaining** water methods based upon similarities in technologies, methods, and procedures. **The same is true for soil samples provided the data are quantitated as both.**

Organic Extractions

1) For sample preparation staff, four LCSs will be prepared, following the 40 CFR Part 136 Appendix A guidelines, either concurrently or over a period of days**, using second source spiking standards, i.e., standards prepared from a source different than the calibration standards, if available**.

2) Precision and accuracy must be demonstrated for all steps of an extraction procedure by the same analyst. The five critical steps are extraction (separatory funnel shake, etc.), KD, nitrogen evaporation or derivatization, bottle up to final volume**, and spike standard additions**.

3) **Separatory funnel extractions require a separate IDOC from continuous liquid-liquid extractions.**

4) **Soxhlet extractions require a separate IDOC from sonication extractions.**

5) For DRO waters, 8015, TN DRO, JP4, and JP8 each require a unique IDOC.

GC/MS Semivolatile and GC/HPLC

1) For GC/HPLC and GC/MS semivolatiles (extractables), each analyst must make four separate **injections of the appropriate second source ICV standard. For semivolatiles, four aliquots of the ICV standard are taken, internal standards are added, and the standards injected. For GC/HPLC, four replicates of the ICV standard are taken, 1:1 dilutions made, and the standards injected**. Statistics can be calculated for precision and accuracy from the injections of these dilution replicates. The performance of an IDOC using the **ICV** standards can count for both water and soil methods.

Criteria used to evaluate replicates prepared from **the ICV standard analyses** are those used to evaluate the **normal** (ICV) **standards**. For methods with no requirement for an ICV, such as CLP, **dilutions of the high level ICAL standard are to be made and** the **acceptance** criteria are 70-130%. Sporadic marginal failures may be allowed in some instances and on a case-by-case basis. For these compounds, criteria of 50-150% are used.

2) Analysis of four extracted LCSs can also be used to demonstrate capability. The analysis of an extractor's IDOC will also count as an analyst's IDOC, but only for the method(s) and matrix approved under the extraction rules.

Inorganic Preparation

1) For mercury waters, analysis of an IDOC by any current method will give the analyst credit for all remaining water methods based upon similarities in technologies, methods, and procedures. The is true for soil samples.

2) For cyanide waters, analysis of an IDOC by any current method will give the analyst credit for all remaining water methods based upon similarities in technologies, methods, and procedures. The same is true for soil samples.

3) For metal waters, **analysis of an IDOC by any current method will give the analyst credit for all remaining water methods based upon similarities in technologies, methods, and procedures. The same is true for soil samples.**

Inorganic Instrumentation

1) For inorganic analyses, a precision and accuracy study can be generated from the analysis of four consecutive dilution analyses of the **second source** ICV standards. These may be collected over a period of days.

2) The performance of an IDOC using the diluted standard can count for both water and soils for mercury**,** cyanide**, and metals,** if the data is quantitated as both.

3) Analysis of four digested LCSs can also be used to demonstrate capability. The analysis of an extractor's IDOC will also count as an analyst's IDOC, but only for the method(s) and matrix approved under the inorganic preparation rules.

Wet Chemistry

1) For wet chemistry analysts, precision and accuracy studies can be generated using the LCS for all methods**, as long as the LCS is from a second source**. Preliminary preparative steps required by a method must be followed for the LCS.

2) Information regarding cyanide IDOCs is given in the inorganics section of this memo.

3) Whenever possible, IDOC credit will be given to multiple methods based upon similar technologies.

On-going Demonstration of Capabilities (ODOC)/ Continuing Demonstration of Capabilities (CDOC)

1) Annually, an analyst must demonstrate continued proficiency for a procedure. This proficiency must be documented and approved following the same approach as the IDOC. The ODOC must be in the analyst's training file to allow continued performance of a test.

2) ODOC credit can be given for the successful completion of a PT sample or QC reference **material**. The sample must be a single blind to count in this category. This credit can be given for all similar technologies for the specific matrix.

3) ODOC credit can be given for the acceptable performance of four consecutive LCSs. **The successful performance of a Limit of Quantitation Study (LOQ) can be used to satisfy the ODOC requirement .** ODOC credit can be given for multiple methods using the rules defined for an IDOC.

4) **For extractions, ODOC credit is needed for each step of an extraction procedure. The five critical steps are the same as those stated in the IDOC procedure**.

5) **On-going Demonstrations of Capability are commonly referred to as Continuing Demonstrations of Capability**.

6) **Department of Defense (DoD) criteria must be met to satisfy ODOC/CDOC requirements for DoD methods.**

The QA department maintains summary tables of IDOC and ODOC information. Copies are currently available upon request. The supervisors and analysts are responsible for satisfying these requirements, performing these tests in a timely manner, and providing the data to the QA department.

Appendix C

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SOP#	Title
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5.2	Resolution of Complaints and Inquiries for Commercial Clients
5.3	Use and Selection of Subcontract Laboratories
5.5	Subcontract Chain of Custody
5.7	Client Requested Customized Electronic Deliverables
5.8	Creating the Gray Folder
5.13	Client Setup, Creating Projects, Sample Receipt and Invoicing in Element
8.1	Diazomethane Generation
8.2	Sonication Procedure
8.4	Training for Sample Preparation Personnel (Organics)
8.5	Pesticide & Semivolatile Lot Checks

SOP#	Title
9.1	Verifying Automatic Pipette Calibrations
9.5	Training Procedures for Inorganic Sample Preparation Personnel
9.10	Distillation of Acids
10.1	Preparing Glassware for the Organics Sample Preparation Laboratory (Organics, Acid B/N Extractables, and Pesticides/PCBs)
10.2	Preparing Glassware for the Inorganics Laboratory
10.4	Cleaning Procedure for the Zero Headspace Extractor (ZHE) and Associated Glassware
11.1.1	Preparing Glassware for Volatile Sample Preparations
11.2.1.3	Training for GC/HPLC Personnel
11.2.2.3	Naming Conventions
11.2.2.9	Sample Quantitation
11.2.2.11	Training Procedures for Semivolatile GC/MS Personnel
11.3.1.4	Instrument Maintenance Procedures
11.3.1.5	Performing Quarterly and Annual Verification Studies of Method and Instrument Parameters for Inorganics
11.3.2.1	Wavelength Calibration Check for Spectronic 21D Spectrophotmeter
12.1	Hazardous Waste Disposal
12.2	Spill Control and Cleanup
13.1	Creating New Standard Operating Procedures
13.2	Revising Standard Operating Procedures
13.4	Numerical Data Reduction
13.5	Assessing Blank Water Purity
13.6	Proper Documentation Procedures
13.11	Performing Organic Method Detection Limit and Verification Studies
13.12	Equipment Maintenance Records and Tag-Out Procedures
13.13	Creating and Updating Control Limits
13.15	Documenting Refrigerator and Freezer Temperatures
13.16	Top-Loading Balance Calibration and Maintenance
13.17	Analytical Balance Calibration Check and Maintenance
13.18	Manual Chromatographic Peak Integration Procedures
13.19	Traceability of Purchased and Prepared Standard Reference Material, Solvents and Reagents
14.1.1.2	ThruPut Method Creation
14.1.1.12	Data Review Procedures for Volatile CLP Reports
14.2.1.1	GC Data Review for CLP
14.2.1.2	GC Data Review for SW-846
14.2.1.3	Thru-Put* Method Creation and Verification (Gilbert: Target 3.4)
14.2.1.4	GC Data Review for SOM01.1
14.2.2.8	Data Review Procedures for Semivolatile CLP Reports
14.2.2.9	Semivolatile Case Review
14.2.2.10	Thru-Put* Method Creation and Verification (Einstein: Target 3.5)
14.3.1.2	Data Review of Inorganic Cases/SDGs for EPA/CLP, SW846, and Commercial Non-CLP
14.3.1.3	Case Review of SW-846 and Commercial Non-CLP
14.3.1.4	Case Review of EPA and non-EPA CLP Cases
14.3.1.6	Using MARRs Software to Produce EPA CLP Data Package Deliverables and Electronic Data Deliverables (EDDs)
14.3.2.1	Data Management: Wet Chemistry Data Review and Verification
15.1	Data Security
15.2	Software Documentation

SOP#	Title
15.4	System Backup and Archival for all UNIX and Windows-based Computer Systems
15.5	Assigning User Classes in Promium Element
15.13	Software Validation
16.1	Hiring, Training, and Promotion Qualifications Program
17.1	SOP Review, Distribution, and Document Control
17.2	Corrective Action Reports (CARs), Preventative Action Reports (PARs), and Documenting Exceptions
17.3	Quality Assurance Audits
17.4	Performing an Audit Trail/ThruPut/Target Audit
17.6	Performing an Audit of Sample Storage and Chain-of-Custody Procedures
17.7	Performing an Annual Audit of Safety Practices and Equipment
17.8	Performing a Reagent (Solvent and Chemical) and Standard Storage and Control Audit
17.10	Performing a Logbook and Record-Keeping Audit
17.12	Calibrating Laboratory Temperature Measurement Devices
17.13	Corrective Action Response Procedures for EPA CLP Evidence Audit Reports
17.14	Processing Proficiency Test (PT) Samples
17.16	Precision, Accuracy, and Estimation of Measurement Uncertainty in the Laboratory
17.17	Document Control
17.18	Process for Product Development
17.19	Ethical Conduct and Data Integrity
17.20	Compiling Electronic Data for Data/Tape Audit Submissions to the U.S. Environmental Protection Agency (EPA)

Appendix D

Certification Summary Table

Accrediting Body	ID Number	Type
Connecticut Department of Public Health	PH-0522	State
Florida Department of Health	E87047	Primary NELAP accreditation
State of Illinois Environmental Protection Agency	200034	Secondary NELAP accreditation
State of Louisiana Department of Environmental Quality	AI 8366 03071	Secondary NELAP accreditation
Kansas Department of Health and Environment	**E-10392**	**Secondary NELAP accreditation**
New Jersey Department of Environmental Protection	NC249	Secondary NELAP accreditation
New York Department of Health	10065	Secondary NELAP accreditation
North Carolina Department of Environment, and Natural Resources	79	Home state certification
Ohio EPA Voluntary Action Program (VAP)	CL0060	State
Pennsylvania Department of Environmental Protection	68-00672	Secondary NELAP accreditation
South Carolina Department of Health and Environmental Control	99055002	State
Wisconsin Department of Natural Resources	999314910	State
Department of **Defense ELAP accredited through Laboratory Accreditation Bureau**	**L2251**	Agency
U.S. EPA Contract Laboratory Program (CLP)	LIBRTY	Agency
National Environmental Laboratory Accreditation Program (NELAP)	E87047	NELAP accreditation based upon FLDOH primary accreditation. Accepted by states marked on this **table,** OR, UT and the non-NELAC states: GA, ME, VT, and WA
U.S. Department of Agriculture	P330-09-00066	Permit to receive soil from foreign sources

Appendix E

NORTH CAROLINA
Department of The Secretary of State

To all whom these presents shall come, Greetings:

I, ELAINE F. MARSHALL, Secretary of State of the State of North Carolina, do hereby certify the following and hereto attached to be a true copy of

ARTICLES OF INCORPORATION

OF

LIBERTY ANALYTICAL CORPORATION

the original of which was filed in this office on the 16th day of July, 1996.

IN WITNESS WHEREOF, I have hereunto set my hand and affixed my official seal at the City of Raleigh, this 6th day of November, 2008

Elaine F. Marshall

Secretary of State

Certification# 88721335-1 Reference# 9477123-ea Page: 1 of 2
Verify this certificate online at www.secretary.state.nc.us/verification

Appendix E (Continued)

0-040460

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JUL 16 1996
JANICE H. FAULKNER
SECRETARY OF STATE
NORTH CAROLINA

EFFECTIVE

ARTICLES OF INCORPORATION
OF
715 CORP.

The undersigned hereby submits these Article of Incorporation for the purpose of forming a business corporation under the laws of the State of North Carolina:

1. The name of the corporation is: 715 Corp.

2. The number of shares the corporation is authorized to issue is 100,000 of common stock.

3. The address of the initial registered office of the corporation in the State of North Carolina is 4300 Six Forks Road, Suite 700, Raleigh, Wake County, North Carolina 27609; and the name of its initial registered agent at such address is: Stephani W. Humrickhouse.

4. The name and address of the incorporator are: Stephani W. Humrickhouse, 4300 Six Forks Road, Suite 700, Raleigh, NC 27609.

5. To the fullest extent permitted by the North Carolina Business Corporation Act as it exists or may hereafter be amended, no person who is serving or who has served as a director of the corporation shall be personally liable to the corporation or any of its shareholders for monetary damages for breach of duty as a director. No amendment or repeal of this article, nor the adoption of any provision to these Articles of Incorporation inconsistent with this article, shall eliminate or reduce the protection granted herein with respect to any mattery that occurred prior to such amendment, repeal, or adoption.

6. These article will become effective upon filing.

This the 15th day of July, 1996.

Stephani W. Humrickhouse
STEPHANI W. HUMRICKHOUSE INCORPORATOR

Appendix E (Continued)

NORTH CAROLINA
Department of The Secretary of State

To all whom these presents shall come, Greetings:

I, ELAINE F. MARSHALL, Secretary of State of the State of North Carolina, do hereby certify the following and hereto attached to be a true copy of

ARTICLES OF AMENDMENT

OF

LIBERTY ANALYTICAL CORPORATION

the original of which was filed in this office on the 17th day of September, 1996.

IN WITNESS WHEREOF, I have hereunto set my hand and affixed my official seal at the City of Raleigh, this 6th day of November, 2008

Elaine F. Marshall

Secretary of State

Certification# 88721336-1 Reference# 9477123-ea Page: 1 of 2
Verify this certificate online at www.secretary.state.nc.us/verification

Appendix E (Continued)

0-044146

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Sep 17, 1996

ARTICLES OF AMENDMENT

96 261 9064

EFFECTIVE
JANICE H. FAULKNER
SECRETARY OF STATE

Pursuant to Statute 55-10-06 of the General Statutes of North Carolina, the undersigned corporation hereby submits the following Articles of Amendment for the purpose of amending its Articles of Incorporation:

1. The name of the corporation is: 715 Corp.

2. The following amendment to the Articles of Incorporation of the Corporation was duly adopted by unanimous consent of the Board of Directors and Shareholder of the Corporation on the 28th day of August.

The name of the Corporation is changed to Liberty Analytical Corporation. There are no other changes to the Articles of Incorporation.

3. These Articles will be effective upon filing.

This the 5th day of September, 1996.

715 CORP.

By: _____
William R. Gates, President

N:\C.11.C:\LIBERTY\AMEND.ART

Certification# 88721336-1 Reference# 9477123-ea Page: 2 of 2

ANALYTICAL SOPs

SOP 1.1.3.1

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 178 SOP Section #: 1.1.3.1 Revision #: 13

SOP Title: Preparation and GC Analysis of Gasoline Range Organics (GRO) in Aqueous Samples by Purge and Trap Following SW-846, CA LUFT and TN GRO

Effective date: (QA fills in) 2-16-11

- Procedure prepared by: [signature] Date: 2-16-11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Patricia A Murphy Date: 2-16-11
- Reason for change: Annual review and Addition of DoD procedures and protocols
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition Update III, Method 8015B CALUFT May 1988; TN GRO; Department of Defense Quality Systems Manual for Environmental Laboratories Version 4.2 October 25, 2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 178: Preparation and GC Analysis of Gasoline Range Organics (GRO) in Aqueous Samples by Purge and Trap Following SW-846, CA LUFT, and TN GRO

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Instrument Procedure 178: Preparation and GC Analysis of Gasoline Range Organics (GRO) in Aqueous Samples by Purge and Trap Following SW-846, CA LUFT, and TN GRO

1.0 Scope and Application

This method is used to detect purgeable total petroleum hydrocarbons (TPH), including gasoline range organics (GRO), in aqueous samples by GC/FID analysis using Method 8015B and the purge and trap technique in Method 5030B. GROs correspond to the range of alkanes from C_6 to C_{10} and covering a boiling point range of approximately 60 ^{o}C – 170 ^{o}C. Qualitative and quantitative data are generated using an FID detector. This standard operating procedure (SOP) also describes the analytical requirements for the California (CA) LUFT and Tennessee (TN) methods.

The reporting limit is the low level calibration standard concentration. The reporting limits are as follows:

- Methods 8015B and CA LUFT – 0.50 mg/L
- Method TN VPH – 1.0 mg/L

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies. Refer to the Quality Manual for procedures for performing and documenting demonstration of capability.

2.0 Summary of Method

Five milliliters of sample are aliquotted into a 5 mL syringe and spiked with surrogate solution. Helium is bubbled through the sample contained in a specially designed purge chamber at ambient temperature. The target analytes are efficiently transferred from the aqueous phase to the vapor phase. The vapor is then swept through a sorbent trap where the target analytes are trapped. After purging is completed, the trap is heated and back-flushed with helium to desorb the target analytes onto a GC column. The GC is temperature programmed to separate the target compounds which are detected by flame

ionization detector (FID). The FID, which is generally non-selective and responsive to all organic compounds, is used to sum the areas of all the peaks over a given range.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 **Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project

requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.**6** Reporting Units – mg/L

3.**7** A **Sample Delivery Group (**SDG**)** is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DOD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% (10% for SC DHEC). If samples are batched together from different sites, project-specific QC must be processed.

3.**8** FID – a detector that measures the electrical current from ions produced by controlled combustion of chromatographic eluates.

3.**9** GALP – good automated laboratory practices; a quality assurance (QA) measure to document that the computer and data acquisition systems are sufficiently protected from tampering and that any changes made to the data have a documented audit trail.

3.**10** SC DHEC – South Carolina Department of Health and Environmental Control

3.**11** DOD-QSM – Department of Defense Quality Systems Manual

3.12 Extraction Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.13 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank carried through sampling and storage serves as a check of contamination.

4.2 Contamination by carryover is possible when a high concentration sample is analyzed. Any sample analyzed after a high concentration sample should be reanalyzed.

4.3 The vessel that a sample is run in must be verified as clean by running a blank in it or a clean sample previously. Check the blank or sample for the appearance of non-target as well as checking target values.

5.0 Safety

5.1 All samples and standard reference materials must be assumed to be hazardous and must be handled to minimize exposure.

5.2 All samples must be prepared under a laboratory fume hood. Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 Laboratory staff must the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 10 µL gastight syringes, Hamilton 1701N or equivalent

6.2 5 mL gastight syringe, Hamilton or equivalent

6.3 25 µL gastight syringes, Hamilton or equivalent

6.4 50 µL gastight syringe, Hamilton or equivalent

6.5 Gas chromatograph: Varian 3400 **or HP5890**

6.6 Columns: RTX - Volatiles

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6.7 Injection port, not used **for the Varian 3400 GC.** A stainless steel low dead volume connector is used to connect the transfer line from the purge and trap directly to the capillary column**.**

6.8 Detector: Varian FID Detector Model 3400 **or HP FID**

6.9 Purge device: Tekmar ALS 2016 **or Tekmar Solatek 72**

6.10 Trap and desorb device: The Tekmar LSC 2000 sample concentrator **or Tekmar 3100 sample concentrator**

6.11 Data system – Standards and samples are processed using the EZ Chrom® Elite version 2.6.1 data acquisition system, Target® version 12.0.0.124 Quick Forms report generation software from ThruPut Systems, and Promium Element® LIMS

7.0 Reagents and Standards

Refer to the Standards Preparation Logbook **or Promium Element LIMS** for details on preparation of standards used in this procedure.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water – All water used in this procedure must be reagent-grade Type I water with regard to resistivity of > 10 mega ohm-cm (19th and 20th Editions of Standard Methods, Method 1080) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remained of this SOP as reagent water.

7.2 Methanol – Purge and trap grade or equivalent

7.3 Primary 8015B calibration standard at 2.5 mg/mL - used for initial and continuing calibration.

7.3.1 Prepare the Primary 8015B calibration standard by adding 250 µL Restek unleaded gasoline composite standard (50 mg/mL) to a 5 mL volumetric

flask containing purge and trap methanol and adjust to volume. Prepare this standard every six months.

7.3.2 Dilutions are made by the analyst for the working level calibration standards according to the instructions in Section 10.0.

7.4 Second source 8015B calibration verification standard (also used to prepare the LCS, MS and MSD)

7.4.1 Prepare 8015B calibration verification standard at 2.5 mg/mL by adding 1 mL AccuStandard Gasoline Composite Mix standard (5.0 mg/mL) to a 2 mL volumetric flask containing purge and trap methanol and adjust to volume. Prepare this standard every six months.

7.5 TN GRO 5.0 mg/mL calibration standard – used to prepare lowest three points on calibration curve and the continuing calibration standard.

7.5.1 Prepare TN GRO 5.0 mg/mL standard by adding 1 mL AccuStandard TN GRO standard (10,000 µg/mL total components) to a 2 mL volumetric flask containing purge and trap methanol and adjust to volume. Prepare this standard every six months.

7.6 TN GRO 10.0 mg/mL calibration standard purchased as 10 mg/mL from AccuStandard and used to prepare highest two points on calibration curve by diluting as indicated in Section 10.0. Replace this standard every six months.

7.7 Second source TN GRO 5.0 mg/mL standard– Restek Gasoline Component Standard, used to prepare LCS, LCSD, MS, and MSD.

7.7.1 Prepare the Second source TN GRO standard by adding 1 mL Restek Gasoline Component Standard (10mg/mL) to a 2 mL volumetric flask containing purge and trap methanol and adjust to volume. Prepare this standard every six months.

7.7.2 This solution, prepared at the mid-point concentration of the 8015B calibration curve, is used for the 72-hour retention time window study performed for 8015B GRO and as the retention time marker standard analyzed prior to initial and daily calibrations. The standard consists of individual alkane compounds including 2-methylpentane (first eluting compound) and 1,2,4-trimethylbenzene (last eluting compound). (See Attachment 2.)

7.8 GRO Surrogate Spiking Solution, 5.0 mg/mL concentration of 1,4-dichlorobutane

7.8.1 Prepare GRO Surrogate Spiking Solution by adding 500 µL of AccuStandard 1,4-Dichlorobutane (20.0 mg/mL) into a 2 mL volumetric flask containing purge and trap methanol and adjust to volume. Prepare this standard every six months.

7.9 TN Surrogate Spiking Solution at 50 µg/mL

7.9.1 Prepare TN Surrogate Spiking Solution by adding 0.25 mL of AccuStandard p-Isopropyltoluene (2 mg/mL) to a 10 mL volumetric flask containing purge and trap methanol and adjust to volume. Prepare this standard every three months.

7.10 Preparation of the laboratory control sample (LCS), matrix spike and matrix spike duplicate (MS/MSD), and blank are detailed in Section 11.0.

7.10 Preparation of the initial and continuing calibration standards are detailed in section 10.0.

7.11 Standard Storage Requirements

7.11.1 Standards are stored in the freezer at –10 ^{o}C to -20 ^{o}C.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 If samples are preserved, the holding time is 14 days from verified time of sample collection. If samples are unpreserved, the holding time is 7 days.

8.3 Always check and report sample pHs.

9.0 Quality Control

9.1 Surrogates

9.1.1 Surrogate recoveries must be within 40 to 160% for all samples, blanks, LCS, matrix spikes, and matrix spike duplicates.

9.1.2 If surrogate recovery is outside this control range, the sample must be re-analyzed.

9.1.3 To meet the requirements of the DOD-QSM, if the surrogate recovery control limits in 9.1.1 are not met and sufficient volume remains, re-prepare and reanalyze the sample. If obvious chromatographic interference with the surrogate is present, re-analysis may not be necessary.

9.1.3.1 If field sample results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "J" flag.

9.1.3.2 If QC samples results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "Q" flag.

9.2 Method Blanks

9.2.1 Method blanks are prepared and analyzed with every 12 hour calibration period or batch. The method consists of a matrix similar to that of its associated samples that is free from the analytes of interest. The method blank is processed with the same procedures under the same conditions as the associated samples.

9.2.2 The concentration of analyte in the blank cannot exceed the reporting limit.

9.2.3 To meet the requirements of the DOD-QSM, the concentration of analyte in the method blank must not exceed one half the reporting limit.

9.2.4 If the method blank fails the acceptance criteria in sections 9.2.2 or 9.2.3, the source of the problem must be investigated and appropriate corrective action taken. All samples processed with a method blank that does not meet acceptance criteria must be reanalyzed.

9.2.5 Any concentration present in the blank that falls between the MDL and the reporting limit is reported as an estimate and flagged with a "J". Analyte that is detected in the blank and associated samples is qualified with a "B" flag on the report form and discussed in the SDG narrative.

9.2.6 For project specific QC sample requirements for Ohio Voluntary Action (VAP) projects, refer to the Laboratory Information Management System (LIMS).

9.3 Laboratory Control Sample (LCS)

9.3.1 The LCS is a matrix free from the analytes of interest spiked with verified known amounts of analytes. For this procedure, the LCS is reagent water

fortified with the gasoline spiking solution and surrogate. The spiking solution is from a different source than that used to prepare the calibration curve and therefore, serves to verify the calibration curve as well as to determine the accuracy of the method. The LCS is prepared with every 20 samples or calibration batch, whichever is more frequent.

9.3.2 The results of the LCS are used to confirm that sample matrix interference and in the MS/MSD analyses and that the analytical system is in control. The results are also used to indicate problems with the calibration curve. The LCS must meet criteria or the entire batch must be repeated.

9.3.3 LCS recovery limits are 75 to 125% for all methods.

9.3.3.1 For TN GRO, a LCS duplicate (LCSD) is also required. The RPD criteria are ≤ 20%.

9.3.3.2 The LCS/LCSD must meet criteria or the entire batch must be repeated.

9.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

9.4.1 A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix per each sample delivery group, if requested by the client.

9.4.2 The percent recovery criteria of the matrix spikes are 75 to 125% and the RPD criteria are ≤ 20%.

9.4.3 To meet the requirements of the DOD-QSM, the MS/MSD spike recoveries must be assessed using the LCS control limits and the RPD must be ≤ 30%.

9.4.3.1 If the MS/MSD acceptance criteria in section 9.4.2 are not met, the client must be contacted for guidance.

9.4.3.2 Any analyte concentration detected the original un-spiked sample associated with MS/MSD data that does not meet acceptance criteria must be qualified in the SDG narrative as an estimated concentration. Refer to the DOD-QSM "J" flag.

9.4.4 When recoveries do not meet the acceptance criteria or interferences preclude proper assessment of the data, results of the LCS are evaluated to verify that the analytical system is in control.

9.4.5 If surrogate recovery in the original un-spiked sample is not within acceptance limits and the MS/MSD surrogate recoveries are within limits, the original sample should be reanalyzed. If the original sample and the MS/MSD analyses yield the same unacceptable surrogate recoveries, the sample does not require reanalysis.

9.5 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of the State of North Carolina DENR. The MS/MSD fulfills this requirement.

9.6 Retention Time Window Study

9.6.1 A retention time window study is conducted over a 72-hour period. Three analyses of the TN GRO standard, standard are performed and the mean and standard deviation of the retention time of the first eluting component 2-methypentane and the last eluting component 1,2,4-trimethylbenzene are calculated. The study is performed once per year or after major instrumentation maintenance such as column change occurs.

9.6.2 The retention time window is established, prior to performing initial calibrations and daily calibration verifications. Subtract 3 standard deviations of the mean retention time (determined from the current 72-hour retention time window study) from the elution time of the first component 2-methypentane in the retention time marker standard and add 3 standards deviations of the mean retention time to the elution time of the last component 1,2,4-trimethylbenzene in the retention time marker standard. The data associated with the analysis of the retention time marker is provided in the report.

9.6.3 The retention time window derived in section 9.6.2 is applied to the analysis of the continuing calibration verification standard and all subsequent field and QC sample analyses during the analytical sequence.

10.0 <u>Calibration and Standardization</u>

10.1 Initial Calibration

10.1.1 Before analyzing samples and required blanks, establish the retention time range with the 20mg/L TN GRO standard (See 10.1.3) and calibrate each

instrument at five concentrations to determine the instrument sensitivity and linearity of the GC/FID response for the purgeable target compounds.

10.1.2 All standards are prepared by spiking the appropriate volume of the gasoline standard and 1,4-dichlorobutane surrogate for 8015B GRO (or p-isopropyltoluene surrogate for TN GRO) into gastight syringes containing 5 mL of sparged reagent water. All of the primary analytical standards are stored in a freezer when not in use.

10.1.3 Five levels of working standards are prepared for the calibration curve by the analyst using a 2.5 mg/mL gasoline standard for 8015B GRO and a 5.0 and 10 mg/mL gasoline standards for TN GRO.

Calibration Curve Standards – 8015B GRO

Amount of 2.5 mg/mL std. added	**Concentration**	**On-column amount**
1 µL	0.5 mg/L	2.5 µg
2 µL	1.0 mg/L	5.0 µg
10 µL	5.0 mg/L	25.0 µg
20 µL	10.0 mg/L	50.0 µg
50 µL	25.0 mg/L	125.0 µg

Calibration Curve Standards - TN GRO Table

Amount of 10 mg/mL std. (for 2 highest) & 5 mg/mL std. (for all others) added	**Concentration**	**On-column amount**
1 µL	1.0 mg/L	5.0 µg
5 µL	5.0 mg/L	25.0 µg
10 µL	10.0 mg/L	50.0 µg
10 µL	20.0 mg/L	100.0 µg
20 µL	40.0 mg/L	200.0 µg

10.1.4 Surrogate solution is added to each standard at varying concentrations. The surrogate levels are listed below.
Surrogates for Calibration – 8015B GRO

Standard Concentration	**Amount of 5.0 mg/mL surrogate added**	**Concentration**	**On-column amount**

Standard Concentration	**Amount of 5.0 mg/mL surrogate added**	**Concentration**	**On-column amount**
0.5 mg/L	1 µL	1.0 mg/L	5.0 µg
1.0 mg/L	2 µL	2.0 mg/L	10.0 µg
5.0 mg/L	5 µL	5.0 mg/L	25.0 µg
10.0 mg/L	10 µL	10.0 mg/L	50.0 µg
25.0 mg/L	20 µL	20.0 mg/L	100.0 µg

Surrogates for Calibration - TN GRO

Standard Concentration	**Amount of 50.0 µg/mL surrogate added**	**Concentration**	**On-column amount**
1.0 mg/L	1 µL	0.01 mg/L	0.05 µg
5.0 mg/L	5 µL	0.05 mg/L	0.25 µg
10.0 mg/L	10 µL	0.1 mg/L	0.5 µg
20.0 mg/L	20 µL	0.2 mg/L	1.0 µg
40.0 mg/L	25 µL	0.25 mg/L	1.25 µg

10.1.5 The percent Relative Standard Deviation (%RSD) of the calibration factors for the initial calibration must be less than or equal to 20% (25% for TN GRO) for all analytes. If the %RSDs over the five levels is ≤20% (25% for TN GRO), then the mean calibration factor (CF) can be used to calculate the gasoline or other petroleum hydrocarbons. (See Method 8000, section 7.4.2.2.)

10.1.6 If the %RSD of the calibration factors is >20% (25% for TN GRO), a least squares regression of the calibration curve must be used. If the correlation coefficient of the calibration curve is ≥ 0.995, the initial calibration is acceptable and is used to calculate analyte concentration. The intercept of the calibration curve must not be forced through the origin, but must be calculated using the five data points.

10.1.7 If the acceptance criteria in sections 10.1.5 and 10.1.6 are not met, the following corrective actions are preformed. After the problem is identified and corrected, a new initial calibration must be analyzed.

10.1.7.1 If CFs have random variation, check for leaks in the purge vessels.

10.1.7.2 If CFs increase with concentration, check for active sites in the chromatographic system.

10.1.7.3 If CFs from direct injections remain consistent, the active sites are probably in the purge and trap system.

10.1.7.4 If CFs decrease with concentration, check for contamination of the system by analyzing a blank.

10.1.8 The calibration curve is verified using a second source standard. For this method, the LCS serves this purpose and is prepared following the preparation procedure in **Section** 11.1.3.

10.1.8.1 To meet the requirements of the DOD-QSM, a second source initial calibration verification (ICV) standard must be analyzed after the initial calibration.

10.1.8.2 The analyte recovery in the ICV must be ± 20% of that in the initial calibration.

10.1.8.3 If the ICV fails, reanalyze it. If the reanalysis fails, analyze a new initial calibration.

10.2 Continuing Calibration Verification (CCV)

10.2.1 A continuing calibration verification standard must be analyzed, after establishing the retention time range with the 20mg/L TN GRO standard. A 5 ppm gasoline standard is used as the CCV standard.

10.2.2 Each calibration factor in the CCV must be ≤ 15% difference (%D) (10%D for CA LUFT and 25%D for TN GRO) when compared to the mean calibration factor of the most recent acceptable initial calibration.

10.2.3 If the %D exceeds 15% (10% for CA LUFT and 25% for TN GRO), then corrective action must be taken and the CCV re-analyzed. If the re-analysis fails, a new initial calibration must be analyzed.

10.2.4 Once the CCV standard and method blank meets criteria, samples are run and subsequently an acceptable continuing calibration must be analyzed within twelve hours of the injection time of the daily continuing calibration. Additional continuing calibration standards can be analyzed within this twelve-hour period to bracket sample analyses.

10.2.4.1 To meet the requirements of the DOD-QSM, an acceptable CCV standard must be analyzed every 10 samples and at the end of the analytical sequence

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 QC and Field Sample Preparation

11.1.1 Thoroughly mix the sample before aliquotting. Aliquot 5 mL of sample into 5 mL syringe and spike with 5 µL of a 5.0 mg/mL surrogate solution of 1,4-dichlorobutane for 8015 GRO. For TN GRO add 10 **µL** of a 50 µg/mL surrogate solution of p-isopropyltoluene.

11.1.2 Prepare a method blank by filling a 5 mL gastight syringe with 5 mL of reagent water and add the appropriate the surrogate solution. The method blank is analyzed before samples and after the continuing calibration.

11.1.3 Prepare a LCS by filling a 5 mL gastight syringe with 5 mL of reagent water, appropriate surrogate solution, and 10 µL of a 2.5 mg/mL 8015B second source standard. Add 10 µL of a 5.0 mg/mL TN GRO second source standard for TN GRO. Prepare a duplicate LCS, for TN GRO.

11.1.4 Prepare the MS/MSD by taking two 5 mL aliquots of the designated sample and adding the appropriate surrogate solution and 10 µL of a 2.5 mg/mL of 8015B second source standard. Add 10 µL of the 5.0 mg/mL TN GRO second source standard for TN GRO.

11.2 Purge and trap analysis

11.2.1 **For ALS 2016 the s**tandards and samples are loaded into the purge vessel of **auto sampler** through the valve at the top of the purge vessel. **For Tekmar Solatek 72 an aliquot of the standards or samples are transferred into a 40 mL voa vial and loaded into the auto sampler tray.**

11.2.3 The ALS 2016 **auto sampler** is controlled by the Tekmar LSC 2000 **(Sections 11.2.3.1, 11.2.3.2 and 11.2.3.3). The Tekmar Solatek 72 auto sampler controls the Tekmar 3100 (Sections 11.2.3.4, 11.2.3.5 and 11.2.3.6)**.

11.2.3.1 On the front panel of the LSC 2000, push the "F2" button for ALS.

11.2.3.2 For sequential vessels, arrow to the "Start" position and press "Enter." Enter the position number for the first analysis and press "Enter." Use the arrow key to go to the last position ("Stop"). Use the "Enter" key to enter the vessel number for the last analysis. Push the "F2" key and check that the enable schedule is "N" for "No.". Push "F4" two times to exit.

11.2.3.3 To schedule samples to run in vessels that are not in sequential order, push the "F2" button for "Schedule." Answer "Enable schedule?" with "Y" for "Yes". Arrow to each start and stop position and enter the numbers by pressing "Enter", the number then "Enter" again. Press "F4" to exit when complete.

11.2.3.4 On the hand held control panel of the Tekmar Solatek 72, press the "Sched" button.

11.2.3.5 For sequential vessels, press enter to get to the "Start" position. Enter the position number for the first analysis and press "Enter." Then enter the number for the last analysis and press "Enter" again.

11.2.3.6 Press the "Sched" button then "C" to run/updated schedule. Next press "A" to view the schedule status.

11.3 Purge and Trap Setup

11.3.1 Purge and Trap Conditions

- Standby Temperature — Ambient
- Valve Temperature — 120 °C
- Line Temperature — 120 °C
- Purge Preheat — 40 °C (heated purge only)
- Purge Time — 8 min.
- Desorb Preheat — 175 °C
- Desorb — 4 min. at 240 °C
- Bake — 8 min. at 260 °C

11.4 Purge and trap troubleshooting

11.4.1 Check the purge flow in a suspect position by purging that position on the autosampler while measuring the flow from the vent on the LSC 2000. It should be 36 – 38 mL/min. If there is little flow, check the thin tubing between the autosampler and the vessel for constriction or obstruction.

11.4.2 Varying purge flow rates observed in the vessels between the positions are usually due to incomplete switching of the 6 port valve and adjustments by the service department need to be made.

11.5 GC Conditions

- Carrier gas — Helium
- Injector Temperature — 50 °C
- Initial Temperature — 45 °C
- Initial Time — 5 min.
- Flow Rate — 8.2 mL/min.
- Ramp Rate — 5 °C/min to 100 °C hold for 0.5 min.
  50 °C/min to 220 °C hold for 2.0 min.
- GC Mode — capillary
- Detector temperature — 250° C
- Final hold time — 2.0 min.
- Makeup flow — 30 mL/min.

11.6 Detector Conditions

- hydrogen flow rate — 30 mL/min
- air flow rate — 300 mL/min
- polarizing voltage — 100 V
- Attentuation — 8-10

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction."

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\,100$$

12.3.2 Matrix spikes:

$$\%\,\mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\,100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{\left(Value\ 1\ +\ Value\ 2\right)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \times 100$$

12.7 Calibration factors for GC analysis are calculated by dividing the total peak area in the standard by the total mass injected (in micrograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.8 Quantitation of Results

Positive results in samples are determined using the external standard technique. A processed GC file is obtained. The file contains the sum of the response of individual peaks in the gasoline fuel pattern within the elution of the first and last components in the standard. The mean CF from the initial calibration is used in the calculation.

The concentration of the gasoline is determined by the following formula:

$$\text{Conc. (mg/L)} = \frac{\text{area x dilution}}{\text{CF x volume purged}}$$

No area may be subtracted from the GRO retention time windows in calculating results except the area contributed by the surrogate. In that case, only the area of a surrogate peak "riding" the hydrocarbon envelope may be subtracted. Peak area measured from blanks may not be subtracted from sample area peaks.

12.8.1 Reporting Results for the Modified 8015B CA LUFT Method

12.8.1.1 Integration is performed using the flattest baseline possible from the beginning of the gasoline peak pattern to the end of the peak pattern. Refer to Quality Control SOP 13.18, “Manual Chromatographic Peak Integration Procedures” for guidance in performing manual integrations.

12.8.1.2 Report the presence of significant peaks that are in the gasoline range even if they do not match the standard peak pattern. This should be noted in the SDG narrative when it occurs.

12.8.1.3 Report the presence of significant peaks outside the chromatographic window. Peaks are significant if they can be distinguished above the noise in a chromatogram. Any peak three times the standard deviation of the signal to noise ratio is statistically significant. All peaks (and baseline rises) outside the window are to be reported.

12.9 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 100 µL sample plus 900 µL diluent for a total volume of 1 mL. It should be recorded on the run log as "10x ".

$$Dilution\ factor = \frac{\mu L\ most\ conc.\ extract\ used\ to\ make\ dilution + \mu L\ clean\ solvent}{\mu L\ most\ conc.\ extract\ used\ to\ make\ dilution}$$

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and

controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, Methods 8015A modified, 8015B and 5030B

16.2 Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure. State of California. Leaking Underground Fuel Tank Task Force. May 1988

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 NELAC Standards, **effective July** 2003**, plus revisions**

16.5 EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs), EPA/600/B-07/001, April 2007

16.6 CompuChem Quality Manual, Revision **13**, **November 17, 2009**

16.7 Tennessee Department of Environment and Conservation, UST Division Lab Methods, "Method for Determination of Gasoline Range Organics"

16.8 Department of Defense Quality Systems Manual for Environmental Laboratories, Version **4.2**, **October 25, 2011**

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – Instrument Run Log

17.2 Attachment 2 – Standard Certificate of Analysis

Attachment 1

COMPUCHEM a division of Liberty Analytical Corp DATE ___/___/___ INITIAL TIME OF TUNE_____ SHIFT (A)_____(B)_____
GC VOLATILE RUN LOG TIME TUNE EXPIRES_____ METHOD: 8015A mod.-8015B
COMPUCHEM LOGBOOK 11 G(4) 6 (GC68) SEQUENCE ID:_____ (circle one) or TN GRO

REJECTED	FILE NAME	REPORTED	DATE	TIME	COMPUCHEM ID#	CASE/SDG#	CHEMIST	STD. AMOUNT	WEIGHT GRAMS	COMMENTS (ETC.)	TEKMAR POSITION
			/ /								
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Std. ID: 1,4-Dichlorobutane (Surr.) GRO Std. 2nd Source GRO Std. p-Isopropyltoluene (Surr.) TN GRO Std. 2nd Source TN GRO Std.
Lot #: _____ _____ _____ _____ _____ _____

REVIEWED BY: _____ DATE _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

12/31/08: JR

Note: This page subject to change without notice.

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Attachment 2

AccuStandard Inc.

125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: GRH-002S
DESCRIPTION: Gasoline Standard
LOT #: B2010289
SOLVENT: MeOH

EXPIRATION: On-going Stability Program

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/MS)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
2-Methylpentane	107-83-5	100	1501	1501
2,2,4-Trimethylpentane	540-84-1	99.6	1500	1494
Heptane	142-82-5	100	500.8	500.8
Benzene	71-43-2	100	500.8	500.8
Toluene	108-88-3	100	1501	1501
Ethylbenzene	100-41-4	100	500.8	500.8
m-Xylene	108-38-3	99.7	1000	997
p-Xylene	106-42-3	100	1001	1001
o-Xylene	95-47-6	99.0	1001	991
1,2,4-Trimethylbenzene	95-63-6	97.1	1030 *	1000

10 Components

* Weight compensated to 100% purity

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty $u_c(y)$. It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is $U_c(y)$ *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR ORG/NO 001
Rev. 9/98

SOP 1.1.3.2

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP179 SOP Section #: 1.1.3.2 Revision #: 11

SOP Title: Preparation and Analysis of Gasoline Range Organics (GRO) in Soil/Sediment Samples by Purge and Trap Following SW-846, CA LUFT, and TN GRO

Effective date: (QA fills in) 2-22-11

- Procedure prepared by: _____ Date: 2-22-11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Patricia A Murphy Date: 2-22-11
- Reason for change: Annual review and Addition of DoD procedures and protocols
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Edition Update III, Method 8015B, CA LUFT, May 1988; TN GRO; Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 179: Preparation and Analysis of Gasoline Range Organics (GRO) in Soil/Sediment Samples by Purge and Trap Following SW-846, CA LUFT, and TN GRO

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Instrument Procedure 179: Preparation and Analysis of Gasoline Range Organics (GRO) in Soil/Sediment Samples by Purge and Trap Following SW-846, CA LUFT, and TN GRO

1.0 Scope and Application

This method is used to detect purgeable total petroleum hydrocarbons (TPH), including gasoline range organics (GRO), in soil or sediment samples by GC/FID analysis using Method 8015A modified or Method 8015B. GROs correspond to the range of alkanes from C_6 to C_{10} and covering a boiling point range of approximately 60 ^oC – 170 ^oC. Qualitative and quantitative data are generated using an FID.

The reporting limit is the low level calibration standard concentration. The reporting limits are as follows:

- Modified method 8015A and CA LUFT – 0.50 mg/Kg
- Method 8015B low-level – 0.5 mg/Kg
- Method 8015B medium-level – 25.0 mg/Kg or 5.0 mg/Kg
- TN EPH – 1.0 mg/Kg

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies. Refer to the Quality Manual for procedures for performing and documenting demonstration of capability.

2.0 Summary of Method

2.1 Method 5030/8015A modified and CA LUFT.

Five grams of sample, collected unpreserved in a glass jar, are weighed into a sparging tube **or 40mL voa vial**. The **sparging** tube is attached to the **Tekmar 2016 ALS** autosampler and 5 mL of reagent water spiked with surrogate solution is added. **The 40mL voa vial is placed in the Tekmar Solatek 72 autosampler tray.** Analysis proceeds as outlined in **Section** 2.3.

If it is determined that the sample needs to be diluted, no less than 1 gram of the sample can be analyzed in order to bring the analytes into the upper half of the analytical range.

NOTE: Modified method 8015A is not allowed for samples reported to the SC DHEC.

NOTE: Modified method 8015A is not allowed for samples reported to the Ohio VAP unless they are high level samples.

2.2 Method 5035A/8015B

Samples received in EnCore™ sampling devices are either frozen for low-level analysis or extracted with methanol for medium-level analysis in the laboratory within 48 hours of collection.

For low-level analyses, samples are extruded from the EnCore™ sampler into sparging tubes **or 40mL voa vial**. **The sparging tube is attached to the Tekmar 2016 ALS autosampler, and 5 mL of reagent water spiked with surrogate solution is added. The 40 mL voa vial is placed in the Tekmar Solatek 72 autosampler tray. Analysis proceeds as outlined in Section 2.3.**

For medium-level analyses, 100 to 250 µL (based on client reporting requirements) of methanol extracts (containing surrogate) are added to 5 mL aliquots of reagent water. The samples are injected into sparging tubes that have been connected to the autosampler **or 40 mL voa vial is placed in the Tekmar Solatek 72 autosampler tray.** The analysis proceeds as in **Section** 2.3.

2.3 Once the samples have been prepared and loaded onto the autosampler, analysis proceeds as follows. Helium is bubbled through the sample. The target analytes are efficiently transferred to the vapor phase. The vapor is then swept through a sorbent trap where the target analytes are trapped. After purging is completed, the trap is heated and back flushed with helium to desorb the target analytes onto a GC column. The GC is temperature programmed to separate the target analytes. The analytes are detected by an FID.

The FID, which is generally non-selective and responsive to all organic compounds, is used to sum the areas of all the peaks over a given range.

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3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.**6** Reporting Units – mg/Kg

3.**7** A **Sample Delivery Group (**SDG**)** is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% (10% for SC DHEC). If samples are batched together from different sites, project-specific QC must be processed.

3.**8** Flame Ionization Detector (FID) – a detector that measures the electrical current from ions produced by controlled combustion of chromatographic eluates.

3.**9** GALP – good automated laboratory practices; a quality assurance (QA) measure to document that the computer and data acquisition systems are sufficiently protected from tampering and that any changes made to the data have a documented audit trail.

3.**10** SC DHEC – South Carolina Department of Health and Environmental Control

3.**11** **DoD**-QSM – Department of Defense Quality Systems Manual

3.12 Extraction Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.13 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank carried through sampling and storage serves as a check of contamination.

4.2 Contamination by carryover is possible when a high concentration sample is analyzed. Any sample analyzed after a high concentration sample should be reanalyzed.

4.3 The vessel that a sample is run in must be verified as clean by running a blank in it or a clean sample previously. Check the blank or sample for the appearance of non-target as well as target analytes.

5.0 Safety

5.1 All samples and standard reference materials must be assumed to be hazardous and must be handled to minimize exposure.

5.2 All samples must be prepared under a laboratory fume hood. Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 10 µL gastight syringes, Hamilton 1701N or equivalent

6.2 5 mL gastight syringes, Hamilton or equivalent

6.3 25 µL gastight syringes, Hamilton or equivalent

6.4 50 µL gastight syringes, Hamilton or equivalent

6.5 Top-loading balance, capable of weighing accurately to 0.01 grams

6.6 Column: RTX-Volatiles

6.7 Injection port, not used **for the Varian 3400 GC**. A stainless steel low dead volume connector is used to connect the transfer line from the purge and trap directly to the capillary column.

6.8 Gas chromatograph: Varian 3400 **or HP5890**

6.9 Trap: Supelco Purge Trap J

6.9 Detector: Varian FID Detector Model 3400 **or HP FID**

6.10 Purge device: Tekmar 2016 ALS **or Tekmar Solatek 72**.

6.11 Trap and desorb device: The Tekmar LSC 2000 **sample concentrator or Tekmar 3100 sample concentrator**

6.12 Data system – Standards and samples are processed using the EZ Chrom® Elite data acquisition system, Target® Quick Forms report generation software from ThruPut Systems, and Promium Element® LIMS

7.0 Reagents and Standards

Refer to the Standards Preparation Logbook **or Promium Element LIMS** for details on preparation of standards used in this procedure.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water – All water used in this procedure must be reagent-grade Type I water with regard to resistivity of > 10 mega ohm-cm (19th and 20th Editions of Standard Methods, Method 1080) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remained of this SOP as reagent water.

7.2 Methanol – purge and trap grade

7.3 Primary 8015B calibration standard - used to prepare 8015A modified, 8015B and CA LUFT initial and continuing calibration standards.

7.3.1 Prepare the primary 8015B calibration standard at 2.5 mg/mL by adding 250 µL Restek unleaded gasoline composite standard (50 mg/mL) to a 5 mL volumetric flask containing purge and trap methanol and bring to volume. Prepare this standard every six months.

7.3.2 Dilutions are made by the analyst for the working level calibration standards according to the instructions in Section 10.0.

7.4 Second source calibration verification standard – used to verify the initial calibration and to prepare the low-level LCS, LCSD, MS, and MSD)

7.4.1 Prepare second source calibration verification standard at 2.5 mg/mL by adding 1 mL AccuStandard Gasoline Composite Mix (5.0 mg/mL) to a 2 mL volumetric flask containing purge and trap methanol and bring to volume. Prepare this standard every six months.

7.5 GRO Medium Level LCS Standard

7.5.1 Transfer the 50 mg/mL NSI gasoline standard to a mini-inert vial for use. This standard expires in 6 months or at manufacturer's expiration date, whichever occurs sooner.

7.6 GRO Medium Level Surrogate

7.6.1 Transfer 20 mg/mL AccuStandard 1, 4-Dichlorobutane to a mini-inert vial for use. This standard expires in 6 months or at manufacturer's expiration date, whichever occurs sooner.

7.7 TN GRO 5.0 mg/mL calibration standard – used to prepare lowest three points of the calibration curve and the continuing calibration standard.

7.7.1 Prepare the TN GRO 5.0 mg/mL calibration standard by adding 1 mL AccuStandard TN GRO standard (10 mg/mL total components) to a 2 mL volumetric flask containing purge and trap methanol and bring to volume. Prepare this standard every six months.

7.8 TN GRO 10.0 mg/mL calibration standard – purchased as 10 mg/mL from AccuStandard and used to prepare the highest two points of the TN GRO calibration curve and the retention time window standards for all methods.

7.9 Second source TN GRO 5.0 mg/mL standard – used to prepare the TN GRO LCS, LCSD, MS, and MSD.

7.9.1 Prepare the TN GRO 5.0 mg/mL standard by adding 1 mL TN GRO 10.0 mg/mL standard to a 2 mL volumetric flask containing purge and trap methanol and bring to volume. Prepare this standard every six months.

7.9.2 This solution, prepared at the mid-point concentration of the 8015B calibration curve, is used for the 72-hour retention time window study performed for 8015B GRO and as the retention time marker standard analyzed prior to initial and daily calibrations. The standard consists of individual alkane compounds including 2-methylpentane (first eluting compound) and 1,2,4-trimethylbenzene (last eluting compound). (See Attachment 2.)

7.10 GRO Surrogate Spiking Solution at 5.0 mg/mL

7.10.1 Prepare the GRO Surrogate Spiking Solution by adding 500 µL AccuStandard 1, 4-Dichlorobutane (20.0 mg/mL) in 2 mL volumetric flask containing purge and trap methanol and bring to volume. Prepare this standard every six months.

7.11 TN Surrogate Spiking Solution at 0.05 mg/mL

7.11.1 Prepare the TN Surrogate Spiking Solution by adding 0.25 mL of AccuStandard p-Isopropyltoluene (2 mg/mL) to a 10 mL volumetric flask containing purge and trap methanol and bring to volume. Prepare this standard every three months.

7.12 Preparation of the laboratory control sample (LCS), matrix spike and matrix spike duplicate (MS/MSD), and blank is detailed in Section 11.0.

7.13 Preparation of the initial and continuing calibration standards is given in section 10.0.

7.14 Standard Storage Requirements

7.14.1 Standards are stored in the freezer at –10 ^{o}C to -20 ^{o}C.

8.0 Sample Preservation and Storage

8.1 Samples are preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Soil samples may be collected in unpreserved glass containers for Method 8015A modified. Holding time is 14 days from sample collection.

NOTE: This option is not allowed for Ohio VAP

8.3 Sample received in EnCore™ samplers must be frozen or preserved with methanol within 48 hours from sample collection. Analytical holding time after preservation is 14 days from sample collection.

9.0 Quality Control

9.1 Surrogates

9.1.1 All field and QC samples are spiked with surrogate.

9.1.2 Surrogate recovery criteria are 40 to 160% for all methods.

9.1.3 If acceptance criteria in **Sections** 9.1.2**, 9.2.4, and 9.3.4** are not met, re-analyze the sample.

9.1.4 To meet the requirements of the **DoD**-QSM, if the surrogate recovery control limits in **Section** 9.1.2 are not met**,** and sufficient volume remains, re-prepare and reanalyze the sample. If obvious chromatographic interference with the surrogate is present, re-analysis may not be necessary.

9.1.4.1 If field sample results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "J" flag.

9.1.4.2 If QC samples results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "Q" flag.

9.2 Method Blanks

9.2.1 Method blanks are prepared and analyzed with every 12 hour calibration period or batch of not more than 20 samples. The method consists of a matrix similar to that of its associated samples that is free from the analytes of interest. The method blank is processed with the same procedures under the same conditions as the associated samples.

9.2.2 The concentration of analyte in the blank cannot exceed the reporting limit.

9.2.3 To meet the requirements of the **DoD**-QSM, the concentration of analyte in the method blank must not exceed one half the reporting limit.

9.2.4 If the method blank fails the acceptance criteria in **Sections** 9.2.2 or 9.2.3, the source of the problem must be investigated and appropriate corrective action taken. All samples processed with a method blank that does not meet acceptance criteria must be reanalyzed.

9.2.5 Any analyte concentration present in the blank that falls between the MDL and the reporting limit is reported as an estimate and flagged with a "J". Any analyte that is detected in the blank and associated samples is qualified with a "B" flag on the report form and explained in the SDG narrative.

9.2.6 For project specific QC sample requirements for Ohio Voluntary Action Program (VAP) projects, refer to the Laboratory Information Management System (LIMS).

9.3 Laboratory Control Sample (LCS)

9.3.1 The LCS is a matrix free from the analytes of interest spiked with verified known amounts of analytes. For this procedure, the LCS is Ottawa sand fortified with the gasoline spiking solution and surrogate. The spiking solution is from a different source than that used to prepare the calibration curve and therefore, serves to verify the calibration curve as well as to determine the accuracy of the method. The LCS is prepared with every 20 samples or calibration batch, whichever is more frequent.

9.3.2 The results of the LCS are used to confirm the presence of sample matrix interference in the MS/MSD analyses and that the analytical system is in control.

9.3.3 Control limits for the LCS spike recovery are 75 to 125 for all methods. **If the LCS is used as an ICV the recovery is 80 to 120.**

9.3.4 The LCS must meet the acceptance criteria in **Section** 9.3.3 or the entire batch must be re-prepared and analyzed.

9.3.5 For TN GRO, a LCS duplicate (LCSD) is also required. The RPD criterion is 20.

9.3.5.1 The LCS/LCSD must meet the acceptance criteria in **S**ections 9.3.3 and 9.3.5 or the entire batch must be re-prepared and analyzed.

9.4 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

9.4.1 A matrix spike and matrix spike duplicate must be performed for each group of samples of a similar matrix per each sample delivery group, when requested by the client.

9.4.2 The spike recovery criteria for the MS/MSD are 75 to 160% for all methods. The RPD for all methods is 20.

9.4.3 To meet the requirements of the DOD-QSM, the MS/MSD spike recoveries must be assessed using the LCS control limits and the RPD must be ≤ 30%.

9.4.3.1 If the MS/MSD acceptance criteria in section 9.4.3 are not met, the client must be contacted for guidance.

9.4.3.2 Any analyte concentration detected in the original un-spiked sample associated with MS/MSD that does not meet acceptance criteria must be qualified in the SDG narrative as an estimated concentration. Refer to the DOD-QSM "J" flag.

9.4.4 When recoveries do not meet the acceptance criteria or interferences preclude proper assessment of the data, results of the LCS are evaluated to verify that the analytical system is in control.

9.4.5 If surrogate recovery in the original un-spiked sample is not within acceptance limits and the MS/MSD surrogate recoveries are within limits, the original sample should be reanalyzed. If the original sample and the MS/MSD analyses yield the same unacceptable surrogate recoveries, then the sample does not require reanalysis.

9.5 Duplicates, at a frequency of 5%, are required when processing samples submitted to meet the regulatory requirements of the State of North Carolina DENR. The MS/MSD fulfills this requirement.

9.6 Retention Time Window Study

9.6.1 A retention time window study is conducted over a 72-hour period. Three analyses of the TN GRO standard, standard are performed and the mean and standard deviation of the retention time of the first eluting component 2-methypentane and the last eluting component 1,2,4-trimethylbenzene are calculated. The study is performed once per year or after major instrumentation maintenance such as column change occurs.

9.6.2 The retention time window is established, prior to performing initial calibrations and daily calibration verifications. Subtract 3 standard deviations of the mean retention time (determined from the current 72-hour retention time window study) from the elution time of the first component 2-methypentane in the retention time marker standard and add 3 standards deviations of the mean retention time to the elution time of the last component 1, 2, 4-trimethylbenzene in the retention time marker standard. The data associated with the analysis of the retention time marker is provided in the report.

9.6.3 The retention time window derived in **Section** 9.6.2 is applied to the analysis of the continuing calibration verification standard and all subsequent field and QC sample analyses during the analytical sequence.

10.0 <u>Calibration and Standardization</u>

10.1 Initial Calibration

10.1.1 Before analyzing samples and required blanks, establish the retention time range with the 20 mg/L TN GRO standard. Calibrate each instrument at five concentrations to determine the instrument sensitivity and linearity of the GC/FID response for the purgeable target compounds.

10.1.2 Prepare calibration standards for low-level soil analysis by adding gasoline standard and surrogate to 5 mL gastight syringes containing sparged reagent water. Add the contents of the syringes to glass purge tubes **or**
40 mL voa vials that contain 5 grams of Ottawa sand**,** which are **loaded** to the instrument's autosampler.

10.1.3 Prepare calibration standards for medium-level analyses by adding the a 2.5 mg/mL gasoline standard solution and 5.0 mg/mL surrogate solution to 5 mL gastight syringes containing sparged reagent water and 100 or 250 µL of methanol. Prepare the working standards as follows:

µL of GRO STD Solution	Working STD Concentration	On-column STD Amount
0.5 µL	0.25 mg/L	1.25 µg
2 µL	1.0 mg/L	5.0 µg
10 µL	5.0 mg/L	25.0 µg
20 µL	10.0 mg/L	50.0 µg
50 µL	25.0 mg/L	125.0 µg

µL of Surrogate Solution	Surrogate Concentration	On-column surrogate Amount
0.5 µL	0.5 mg/L	2.5 µg
2 µL	2.0 mg/L	10.0 µg
10 µL	5.0 mg/L	25.0 µg
20 µL	10.0 mg/L	50.0 µg
50 µL	20.0 mg/L	100.0 µg

10.1.4 Five levels of working standards are prepared for the calibration curve by the analyst using a 2.5 mg/mL gasoline standard solution and 5.0 mg/mL surrogate solution for 8015A modified, 8015B and CA LUFT. Prepare the working standards as follows.

µL of GRO STD Solution	Working STD Concentration	On-column STD Amount
1 µL	0.5 mg/L	2.5 µg
2 µL	1.0 mg/L	5.0 µg
10 µL	5.0 mg/L	25.0 µg
20 µL	10.0 mg/L	50.0 µg
50 µL	25.0 mg/L	125.0 µg

µL of surrogate	Surrogate	On-column surrogate

Solution	Concentration	Amount
1 µL	1.0 mg/L	5.0 µg
2 µL	2.0 mg/L	10.0 µg
10 µL	5.0 mg/L	25.0 µg
20 µL	10.0 mg/L	50.0 µg
50 µL	20.0 mg/L	100.0 µg

10.1.5 Five levels of working standards are prepared for TN GRO using the 5.0 and 10 mg/mL gasoline standards solutions and the 0.05 mg/L TN GRO surrogate solution. The 10 mg/mL gasoline standard solution is used to prepare the two high concentration working standards. Prepare the working standards as follows.

µL of TN GRO STD Solution	Working STD Concentration	On-column Amount
1 µL	1.0 mg/L	5.0 µg
2 µL	5.0 mg/L	25.0 µg
10 µL	10.0 mg/L	50.0 µg
10 µL	20.0 mg/L	100.0 µg
20 µL	40.0 mg/L	200.0 µg

µL of TN surrogate Solution	TN surrogate Concentration	On-column Amount
1 µL	0.01 mg/L	0.05 µg
5 µL	0.05 mg/L	0.25 µg
10 µL	0.1 mg/L	0.5 µg
20 µL	0.2 mg/L	1.0 µg
25 µL	0.25 mg/L	1.25 µg

10.1.6 The percent relative standard deviation (%RSD) of the five initial calibration standards must be ≤20% (25% for TN GRO). If this criterion is met, then the mean calibration factors (CF) can be used to calculate the analyte and surrogate concentrations.

10.1.7 If the %RSD is >20% (25% for TN GRO), a least squares regression of the calibration curve must be used. If the correlation coefficient of the calibration curve is ≥ 0.995, the initial calibration curve is acceptable and may be used to calculate analyte and surrogate concentrations. The

intercept of the calibration curve must not be forced through origin, but must be calculated using the five data points.

10.1.8 If the acceptance criteria in **Section** 10.1.6 or 10.1.7 are not met, the following corrective actions are performed. After the problem is identified and corrected, a new initial calibration must be analyzed.

10.1.8.1 If CFs have random variation, check for leaks in the purge vessels.

10.1.8.2 If CFs increase with concentration, check for active sites in the chromatographic system.

10.1.8.3 If CFs from direct injections remain consistent, the active sites are probably in the purge and trap system.

10.1.8.4 If CFs decrease with concentration, check for contamination of the system by analyzing a blank.

10.1.9 The validity of the calibration curve is verified using a second source standard. For this method, the LCS serves this purpose and is prepared following the preparation procedure in Section 11.1.3 following.

10.1.9.1 To meet the requirements of the DOD-QSM, a second source initial calibration verification (ICV) standard must be analyzed after the initial calibration.

10.1.9.2 The analyte recovery in the ICV must be ≤ 20% of that in the initial calibration.

10.1.9.3 If the ICV fails, re-analyze it. If the re-analysis fails, perform corrective actions and analyze a new initial calibration.

10.2 Continuing Calibration Verification (CCV)

10.2.1 A continuing calibration verification standard must be analyzed, after establishing the retention time range with the 20 mg/L TN GRO standard, to start a daily sequence and must meet acceptance criteria in order for sample analysis to proceed. A 5 ppm gasoline standard is used as the CCV standard. Each calibration factor, must be ≤15% difference (%D) (10%D for CA LUFT and 25%D for TN GRO) when compared to the mean calibration factor of the most recent acceptable initial calibration.

10.2.2 If the %D exceeds 15% (10% for CA LUFT and 25% for TN GRO), then corrective action must be taken and the CCV re-analyzed. If the re-analyses fails criteria, a new initial calibration must be analyzed.

10.2.3 Once the daily continuing calibration standard and the method blank meets criteria, samples are run and subsequently an acceptable continuing calibration must be analyzed within twelve hours of the injection time of the daily continuing calibration. Additional continuing calibration standards can be analyzed within this twelve hour period to bracket sample analyses. **If the closing or bracket CCV fails QC criteria, then the entire batch must be reanalyzed.**

10.2.3.1 To meet the requirements of the DOD-QSM, an acceptable CCV standard must be analyzed every 10 samples and at the end of the analytical sequence.

10.3 Balance Calibration

10.3.1 Ensure the balance is calibrated for the day prior to weighing samples. Refer to Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 <u>Procedure</u>

Documentation must follow the requirements in Quality Control SOP 13.6 "Proper Documentation Procedures".

11.1 Method 8015A modified Low-Level Sample Preparation

Note: This option is not allowed for South Carolina Samples **and Ohio VAP**.

11.1.1 Mix sample well, discard any foreign objects such as sticks, stones/rocks, and leaves. Weigh out 5 grams of sample into a sparging tube **or 40 mL voa vial**. Add 5 mL of reagent water spiked with 5 µL of 5.0 mg/mL surrogate solution to the tube a 5 mL gastight syringe. Attach the tube to the instrument's autosampler **or load the voa vial in the autosampler tray**. For TN GRO add 10 µL of a 0.050 mg/mL surrogate solution.

11.1.2 Prepare a method blank by weighing 5 grams of Ottawa sand into a sparging tube **or 40 mL voa vial** and attach **sparging tube** to the instrument's autosampler **or load the voa vial in the autosampler tray**. Add 5 mL of reagent water spiked with 5 µL of 5.0 mg/mL surrogate

solution using a 5 mL gastight syringe. Analyze the method blank before samples and after the continuing calibration. For TN GRO add 10 µL of a 0.050 mg/mL surrogate solution.

11.1.3 Prepare the LCS by weighing 5 grams of Ottawa sand into a sparging tube **or 40 mL voa vial and attach sparging tube to the instrument's autosampler or load the voa vial in the autosampler tray.** Add appropriate surrogate and 10 µL of a 2.5 mg/mL second source gasoline standard. Add 10 µL of a 5.0 mg/mL second source standard for TN GRO. Prepare a duplicate LCS, for TN GRO.

11.1.4 Prepare a MS/MSD using the procedure in **Section** 11.1.1. In addition to surrogate solution, add 10 µL of 2.5 mg/mL second source gasoline standard. Add 10 µL of the 5.0 mg/mL second source standard for TN GRO.

11.2 Method 8015B, CA LUFT, and TN GRO Medium-Level Sample Preparation

Note: The TN GRO method requires 10 g of soil and 10 mL of methanol for the extraction.

11.2.1 Prepare sample extracts by weighing 5.0 grams of sample into 5 mL of methanol in a 40 mL vial. Add 62.5 µL of 20 mg/mL surrogate solution (0.5 mL of 0.050 mg/mL surrogate solution for TN GRO) for a 100 µL extract volume. Shake the sample for 2 minutes, and then allow the soil to settle.

11.2.1.1 For clients that require a 250 µL of extract volume, weigh 5.0 grams of sample into 5 mL of methanol in a 40 mL vial. Add 25 µL of 20mg/mL surrogate solution. Shake the sample for 2 minutes, and then allow the soil to settle.

11.2.2 Prepare a method blank by weighing 5.0 grams of Ottawa sand into 5 mL of methanol in a 40 mL vial. Add 62.5 µL of 20 mg/mL surrogate solution (0.5 mL of 0.050 mg/mL of surrogate solution for TN GRO) for a 100 µL extract volume. Shake the sample for 2 minutes, and then allow the sand to settle

11.2.2.1 For clients that require a 250 µL of extract volume, weigh 5.0 g of sand into 5 mL of methanol in a 40 mL vial. Add 25 µL of 20 mg/mL surrogate. Shake the sample for 2 minutes, and then allow the sand to settle.

11.2.3 Prepare a laboratory control sample by weighing 5.0 grams of Ottawa sand into 5 mL of methanol in a 40 mL vial. Add 62.5 µL of 20 mg/mL surrogate solution (0.5 mL of 0.050 mg/mL surrogate solution for TN GRO) and 50 µL of 25 mg/mL second source standard (0.5 mL of 5 mg/mL second source standard for TN GRO) for a 100 µL of extract volume. Shake the sample for 2 minutes, and then allow the sand to settle.

11.2.3.1 For clients that require a of 250 µL of extract volume, weigh 5.0 g of Ottawa sand into 5 mL of methanol in a 40 mL vial. Add 25 µL of 20 mg/mL surrogate solution (0.5 mL of 0.050 mg/mL surrogate solution for TN GRO), and 20 µL of 50 mg/mL NSI Gasoline Standard. Shake the sample for 2 minutes, and then allow the sand to settle.

11.2.4 For duplicate matrix spikes, follow steps in **Section**11.2.3 and use 5.0 g of sample rather than Ottawa sand.

11.2.5 Prepare a 100 µL or 250 µL aliquot of the methanol extract for analysis by purge and trap by using the following procedure.

11.2.5.1 Add 100 µL of methanol extract to 4.9 mL of reagent water in a 5 mL gastight syringe.

11.2.5.2 Add 250 µL of methanol extract to 4.75 mL of reagent water in a 5 mL gastight syringe.

11.2.6 Heated purge is required for low-level soil methods 8015A modified and 8015B. This is accomplished by placing a thermal sleeve on the purge vessel during sample purging.

11.3 Purge and trap analysis

11.3.1 **For ALS 2016 the standards and samples are loaded into the purge vessel of autosampler. For Tekmar Solatek 72 an aliquot of the standards or samples are transferred into a 40 mL voa vial and loaded into the auto sampler tray.**

11.3.2 **The ALS 2016 auto sampler is controlled by the Tekmar LSC 2000 (Sections 11.3.2.1, 11.3.2.2, 11.3.2.3, and 11.3.2.4). The Tekmar Solatek 72 auto sampler controllers the Tekmar 3100 (Sections 11.3.2.5, 11.3.2.6, and 11.3.2.7).**

11.3.2.1 On the front panel of the LSC 2000, push the "F2" button for ALS.

11.3.2.2 For sequential vessels, arrow to the "Start" position and press "Enter." Enter the position number for the first analysis and press "Enter." Use the arrow key to go to the last position ("Stop"). Use the "Enter" key to enter the vessel number for the last analysis. Push the "F2" key and check that the enable schedule is "N" for "No.". Push "F4" two times to exit.

11.3.2.3 To schedule samples to run in vessels that are not in sequential order, push the "F2" button for "Schedule." Answer "Enable schedule?" with "Y" for "Yes". Arrow to each start and stop position and enter the numbers by pressing "Enter", the number then "Enter" again. Press "F4" to exit when complete.

11.3.2.4 Begin sample analysis by pressing the "start" button on the autosampler.

11.3.2.5 On the hand held control panel of the Tekmar Solatek 72, press the "Sched" button.

11.3.2.6 For sequential vessels, press enter to get to the "Start" position. Enter the position number for the first analysis and press "Enter." Then enter the number for the last analysis and press "Enter" again.

11.3.2.7 Press the "Sched" button then "C" to run/updated schedule. Next press "A" to view the schedule status.

11.4 Purge and Trap Setup

11.4.1 Purge and Trap Conditions

- Standby Temperature Ambient
- Valve Temperature 120 °C
- Line Temperature 120 °C
- Purge Preheat 40 °C (heated purge only)
- Purge Time 8 min.

- Desorb Preheat 175 °C
- Desorb 4 min. at 240 °C
- Bake 8 min. at 260 °C

11.5 Purge and trap troubleshooting

11.5.1 Check the purge flow in a suspect position by purging that position on the autosample while measuring the flow from the vent on the LSC 2000. It should be 36 – 38 mL/min. If there is little flow, check the thin tubing between the autosampler and the vessel for constriction or obstruction.

11.5.2 Varying purge flow rates observed in the vessels between the positions are usually due to incomplete switching of the 6 port valve and adjustments by the service department need to be made.

11.6 GC Conditions

- Carrier gas Helium
- Injector Temperature 50 °C
- Initial Temperature 45 °C
- Initial Time 5 min.
- Flow Rate 8.2 mL/min.
- Ramp Rate 5 °C/min to 100 °C hold for 0.5 min.
  50 °C/min to 220 °C hold for 2.0 min.
- GC Mode capillary
- Detector temperature 250° C
- Final hold time 2.0 min.
- Makeup flow 30 mL/min.

11.7 Detector Conditions

- hydrogen flow rate 30 mL/min
- air flow rate 300 mL/min
- polarizing voltage 100 V
- Attentuation 8-10

12.0 <u>Data Analysis and Calculations</u>

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction."

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\,100$$

12.3.2 Matrix spikes:

$$\%\,\mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\,100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{\left(Value\ 1\ +\ Value\ 2\right)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \times 100$$

12.7 Calibration factors for GC analysis are calculated by dividing the total peak area in the standard by the total mass injected (in nanograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.7 Quantitation of Results

Positive results in samples are determined using the external standard technique. A processed GC file is obtained. The file contains the sum of the response of individual peaks in the gasoline fuel pattern within the elution of the first and last components in the standard. The mean CF from the initial calibration is used in the calculation.

The concentration of the gasoline is determined by the following formula:

$$\text{Conc. (mg/kg)} = \frac{\text{area } \mathbf{x} \text{ dilution } \mathbf{x} \text{ dry wt. factor}}{\text{CF } \mathbf{x} \text{ weight extracted (purged)}}$$

No area may be subtracted for the GRO retention time windows in calculating results except the area contributed by the surrogate. In that case, only the area of a surrogate peak "riding" the hydrocarbon envelope may be subtracted. Peak area measured from blanks may not be subtracted from sample area peaks.

12.7.1 Reporting Results for the 8015A modified/8015B/CA LUFT Method

Integration is performed using the flattest baseline possible from the beginning of the gasoline peak pattern to the end of the peak pattern. Refer to Quality Control SOP 13.18, “Manual Chromatographic Peak Integration Procedures” for guidance in performing manual integrations.

Report the presence of significant peaks that are in the gasoline range even if they do not match the standard peak pattern. This should be noted in the SDG narrative when it occurs.

12.7.2 Report the presence of significant peaks outside the chromatographic window. Peaks are significant if they can be distinguished above the noise in a chromatogram. Any peak three times the standard deviation

of the signal to noise ratio is statistically significant. All peaks (and baseline rises) outside the window are to be reported.

12.8 The daily retention time window is established using the 20 mg/L TN GRO standard by subtracting the 3SD value, determined from the 72 hour retention time study, from the elution time for 2-methylpentane, and adding the 3SD value to the elution time of 1,2,4-trimethylbenzene.

12.9 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 100 µL sample plus 900 µL diluent for a total volume of 1 mL. It should be recorded on the run log as "10x ."

12.9.1 Dilution factor

$$Dilution\ factor = \frac{\mu L\ most\ conc.\ extract\ used\ to\ make\ dilution + \mu L\ clean\ solvent}{\mu L\ most\ conc.\ extract\ used\ to\ make\ dilution}$$

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Methods 8015A modified, 8015B, 5030B and 5035A.

16.2 Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure. State of California. Leaking Underground Fuel Tank Task Force. May 1988.

16.3 "Method for Determination of Gasoline Range Organics", TN DEC, March 1999

16.4 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.5 NELAC Standards, **effective July** 2003**, plus revisions**

16.6 EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs), EPA/600/B-07/001, April 2007

16.7 CompuChem Quality Manual, Revision **13**, **November 17, 2009**

16.8 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2011

17.0 Attachments as Tables, Diagrams, **and** Flowcharts

17.1 Attachment 1 – **Example** Instrument Run Log

17.2 Attachment 2 – **Example** Standard Certificate of Analysis

Attachment 1

COMPUCHEM a division of Liberty Analytical Corp DATE ___/___/___ INITIAL TIME OF TUNE_____ SHIFT (A)_____(B)_____
GC VOLATILE RUN LOG TIME TUNE EXPIRES_____ METHOD: 8015A mod.-8015B
COMPUCHEM LOGBOOK 11 G(4) 6 (GC68) SEQUENCE ID:_____ (circle one) or TN GRO

REJECTED	FILE NAME	REPORTED	DATE	TIME	COMPUCHEM ID#	CASE/SDG#	CHEMIST	STD. AMOUNT	WEIGHT GRAMS	COMMENTS (ETC.)	TEKMAR POSITION
			/ /								
			/ /								
			/ /								
			/ /								
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Std. ID: 1,4-Dichlorobutane (Surr.) GRO Std. 2nd Source GRO Std. p-Isopropyltoluene (Surr.) TN GRO Std. 2nd Source TN GRO Std.
Lot #: _____ _____ _____ _____ _____ _____

REVIEWED BY: _____ DATE _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

12/31/08: JR

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Attachment 2

AccuStandard Inc.

125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: GRH-002S
DESCRIPTION: Gasoline Standard
LOT #: B2010289
SOLVENT: MeOH

EXPIRATION: On-going Stability Program

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/MS)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
2-Methylpentane	107-83-5	100	1501	1501
2,2,4-Trimethylpentane	540-84-1	99.6	1500	1494
Heptane	142-82-5	100	500.8	500.8
Benzene	71-43-2	100	500.8	500.8
Toluene	108-88-3	100	1501	1501
Ethylbenzene	100-41-4	100	500.8	500.8
m-Xylene	108-38-3	99.7	1000	997
p-Xylene	106-42-3	100	1001	1001
o-Xylene	95-47-6	99.0	1001	991
1,2,4-Trimethylbenzene	95-63-6	97.1	1030 *	1000

10 Components

* Weight compensated to 100% purity

1. All weights are traceable through National Institute of Standards & Technology, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty $u_c(y)$. It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is $U_c(y)$ *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/NC-001
Rev. 9/98

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CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-238 SOP Section #: 1.1.4.1 Revision #: 9

SOP Title: Preparation of Soil/Sediment/Sludge/Non-Aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed System Purge and Trap by SW-846 Methods 5035A and 3585, and EPA CLP SOW Som01.2

Effective date: (QA fills in) 5-26-2011

- Procedure prepared by: Judith Vezzi Date: 05-20-2011
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Meiner Date: 05-26-2011
- Reason for change: Annual Review and addition of UMS Information
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Ed. Update III, 12/96, Methods 8260B and 3585; 7/02, Method 5035A; US EPA CLP SOW Som01.2

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure –238: Preparation of Soil/Sediment/Sludge/Non-aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap by SW-846 Methods 5035A and 3585, and EPA CLP SOW **SOM01.2**

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Sample Preparation Procedure -238: Preparation of Soil/Sediment/Sludge/Non-aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap by SW-846 Methods 5035A and Method 3585, and EPA CLP SOW **S**OM0**1.2**

1.0 Scope and Application

This procedure is used to prepare soil, sediment, and sludge samples for the analysis of volatile organic compounds by the closed-system purge and trap process using GC/MS Method 8260B or EPA CLP SOW **SOM01.2**. The procedure is based on Method 5035A and Method 3585. The prepared sample may also be analyzed for GRO by Method 8015B. Provisions are also included to prepare (and preserve) samples with methanol when higher concentrations of volatiles are present in the sample.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Several options are available when EnCore™ sampling devices are utilized. These incorporate the use of a sodium bisulfate preservative, freezing at a temperature colder than –7 °C, or, for samples containing high levels of target analytes, preserving in methanol.

2.1 Sodium Bisulfate Preservation

A 5 g sample is taken in the field using a disposable EnCore™ sampling device. At the laboratory and within 48 hours of sampling, the sample is weighed into a standard 40 mL volatile vial containing a magnetic stirring bar and 5 mL of a sodium bisulfate solution (0.2 g of sodium bisulfate per gram of sample weighed.) The vial is sealed with a screw-top, PTFE-lined, septum-sealed cap. The sample container is stored at 4 °C ± 2 °C**,** until analysis which must be completed within 14 days of collection.

2.2 Freezing Preservation

A 5 g sample is taken in the field using a disposable EnCore™ sampling device. At the laboratory and within 48 hours of sampling, the contents of the EnCore™ sampling device are placed into a tared, dry, closed-system purge-and-trap vial, re-weighed to obtain the final sample weight, and the vial placed into a freezer maintained at a temperature colder than –7 °C. This option can be performed with or without the addition of 5 mL of reagent water.

For the State of Florida DEP, samples may be frozen to –10 ^{o}C, but only after the entire contents of the sampling device are extruded into the sample analysis vial containing reagent water.

2.3 Methanol Preservation

For screening purposes, to provide a mechanism to determine the concentration of target analytes which may be present at high levels, the contents of another 5 g EnCore™ sampler and placed into a vial containing 5 mL of methanol. For non-aqueous wastes, 1 g of sample is weighed and placed into a vial to which 5 mL of methanol is added.

2.4 Miscellaneous

For some projects, a 25 g EnCore™ sampler may be used and its contents placed in a 2 oz. jar and preserved with methanol.

Depending on specific project requirements, samples may be received by the laboratory already in sealed 40 mL vials, containing a stirring bar and sodium bisulfate solution, or preserved with methanol. In additional cases, samples may be received in closed-system purge-and-trap vials, with or without water.

3.0 Definitions

3.1 A **Sample Delivery Group (**SDG**)** is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.2 GRO – Gasoline Range Organics

3.3 DoD-QSM – Department of Defense Quality Systems Manual

3.4 SC DHEC – South Carolina Department of Health and Environmental Control

3.5 LIMS – Laboratory Information Management System

3.6 Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

4.0 Interferences

4.1 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. A trip blank, carried through sampling and storage, serves as a check of contamination.

4.2 The laboratory, where volatiles are prepared and analyzed, must be completely free of solvents other than methanol. Persons leaving the organic sample preparation laboratory must not enter the volatile laboratory until sufficient time has passed to avoid the introduction of solvent vapors.

5.0 Safety

5.1 The toxicity of each reagent used in this method is not precisely defined so each must be treated as a potential health hazard. Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous.

5.2 When following this procedure, be sure to wear protective gloves, lab coats, safety glasses and work under a hood.

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5.3 Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment **and** Supplies

6.1 Sample containers

6.1.1 Standard 40 mL volatile vial with screw-top, PTFE-lined, septum-sealed cap. Vials are purchased pre-cleaned from certifying vendors.

6.1.2 Purchased, from certifying vendors, standard 40 mL volatile vials with screw-top, PTFE-lined, septum-sealed cap each containing 5 mL of a 20% solution of sodium bisulfate in reagent water and a stir bar (equivalent to 1.0 g of sodium bisulfate and 5 mL of organic-free water).

6.2 Magnetic stirring bar – PTFE or glass coated

6.3 Stainless steel spatulas

6.4 Top-loading balance, capable of weighing accurately to 0.01 g

6.5 5 or 25 g disposable EnCore™ sampling device

6.6 Graduated pipettes capable of delivering 1 – 10 mL

6.7 Glassware – 10 to 100 mL volumetric flasks

6.8 EnCore™ extrusion tool

6.9 Data system – Standards and samples are processed using the EZ Chrom® Elite data acquisition system, Target® Quick Forms report generation software from ThruPut Systems, and Element DataSystem® LIMS computer software by Promium

7.0 Reagents **and** Standards

Refer to the Standards Preparation Logbook or Promium Element LIMS for details on preparation of standards used in this procedure.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are

also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type I water, with regard to resistivity of > 10 megohm-cm (19th and 20th Editions of Standard Methods, Method 1080), which is subsequently purged with the inert gas, nitrogen, and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as reagent water.

7.2 Methanol – purge and trap grade or equivalent

7.3 Sodium bisulfate ($NaHSO_4$) – ACS reagent grade

7.3.1 20% Sodium bisulfate solution – 200 g $NaHSO_4$ in 1000 mL DI water

8.0 Sample Preservation and Storage

Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 A method blank is prepared for every preparation batch following **Sections** 11.2 – 11.4 with a 5 gram portion (or **Section** 11.10, with 1 g for non-aqueous wastes) of a purified solid matrix (Ottawa sand) in place of sample matrix.

9.2 **Sections** 11.3 – 11.5 (or **Sections** 11.9, 11.10 or 11.11), depending on the option exercised) are repeated twice with 5 gram portions of a designated sample and used for the matrix spike (MS) and matrix spike duplicate (MSD) For SW-846 sample batches, an additional 5 gram portion of a purified solid matrix (Ottawa sand) is weighed into a separate sample container for the laboratory control sample (LCS). For non-aqueous wastes, two 1 gram portions of sample are used for the MS/MSD**,** and 1 gram of a purified solid matrix is used for the for the laboratory control sample (LCS).

9.3 Duplicates, at a frequency of 10%, are required when processing samples submitted to meet the regulatory requirements of North Carolina.

9.4 See the associated analytical SOPs for details on the composition, concentration, and spiking levels of surrogates and/or spikes.

10.0 Calibration **and** Standardization

10.1 Balance Calibration

10.1.1 Ensure the balance is calibrated for the day prior to weighing samples. Refer to Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 When the samples have come to room temperature and are ready to be prepared, assemble the designated materials in the hood.

11.2 For each sample, label (with permanent ink typically on white tape) 3, 40 mL vials with the laboratory ID number of the sample.

11.3 Open the 5 g disposable EnCore™ sampling device. Do not discard any supernatant liquid.

11.4 Place the first vial (with cap), containing 5 mL of a 20% sodium bisulfate solution and a magnetic stirring bar, on the top loading balance. Add the contents of the EnCore™ sampler to the vial. Seal the volatile vial with the PTFE-lined screw cap. Read and record weight. The weight is recorded in the EnCore™ Preparation Log Book (Attachment 1).

11.5 Prepare the second, back-up, sample aliquot by repeating **Sections** 11.3 and 11.4.

11.6 Place the third 40 mL vial and cap on the top-loading balance and transfer 5.0 mL ± 0.1 mL of methanol. Record the weight. Transfer the contents of a 5 g EnCore™ sampler to the vial. Seal the vial with the PTFE-lined screw cap and record the weight. Use this sample for screening purposes and medium/high level analysis, if necessary.

11.7 Repeat **Sections** 11.2 – 11.6 until the required number of samples are prepared, completing the worksheet as the preparation is accomplished.

11.8 If, during the addition of a sample to a vial containing the sodium bisulfate solution, effervescing occurs, that sample must be properly discarded. An empty vial is obtained, weighed, the contents of a 5 gram EnCore™ sample added and the vial sealed with a PTFE-lined screw cap and the weight recorded. This

sample must be placed in a freezer at a temperature colder than –7 °C. Pre-testing for effervescence can also be performed using the bottle supplied for percent moisture.

11.9 Sample preservation by freezing, as described in **Section** 11.8, is also a viable option for all samples received in EnCore™ sampling devices. The contents of the EnCore™ sampling devices are placed in a tared, dry, closed-system, purge-and-trap device, re-weighed to obtain the final sample weight, and the vial placed into a freezer maintained at a temperature colder than –7 °C.

11.10 Sample preparation for the dilution of non-aqueous wastes in methanol (See **Section** 2.3). Place a clean, dry 40 mL vial on the top-loading balance. Tare the balance. Carefully weigh out 1 g of sample into the vial. Record the weight. Add 5.0 mL ± 0.1 mL of methanol. Seal the vial with the PTFE-lined screw cap immediately and shake for 2 minutes.

11.11 Samples prepared by this method are now ready for analysis. (See appropriate analytical method SOP). When the set is complete, store the samples in the appropriate volatile GC or GC/MS laboratory refrigerator or freezer until analysis. The refrigerator temperature must be at 4 °C ± 2 °C for sodium bisulfate preserved samples. The freezer temperature must be < –7 °C for the freezing option.

12.0 Data Analysis **and** Calculations

Calculations must be consistent with the Quality Control SOP 13.4, “Numerical Data Reduction”.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible,

laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 U.S. EPA CLP Statement of Work for Organics, Multi-Media, Multi-Concentration, **SOM01.2**

16.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 8260B and Method 3585.

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 NELAC Standards, effective July 2003, plus revisions

16.6 **EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs), EPA/600/B-07/001, April 2007**.

16.7 CompuChem Quality Manual, Revision **13**, **November 17**, 2009 plus revisions

16.8 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **October 25, 2010**.

16.9 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, 7/02, Method 5035A

17.0 Attachments as Tables, Diagrams, **and** Flowcharts

17.1 Attachment 1 – **Example** EnCore™ Preparation Log

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Attachment 1

COMPUCHEM
a Division of Liberty Analytical
EnCore™ Preparation Log
-238 / -243 / -233

Logbook 11 O (4) 12

SDG:_____ Prepared By:_____ Date:_____

	Prep Number	Lab ID	Date	Time	Weight of Jar (g) *	Weight of Jar + Sample (g)	Weight of Sample (g)
1							
2							
3							
4							
5							
6							
7							
8							
9							
10							
11							
12							
13							
14							
15							
16							
17							
18							
19							
20							
21							
22							
23							
24							
25							

Volume of Methanol Added:_____

Lot Numbers of Preservatives: Sodium Bisulfate:_____ Methanol:_____

Reviewed By:_____ Date:_____

*Weight of Jar for Sodium Bisulfate preserved Encores is the sum of the weights of the vial, preservative and stir bar
*Weight of Jar for Methanol preserved Encores is the sum of the weights of the vial and preservative

Note: The insertion of "N/A" for "Volume of Methanol Added" and "Lot Numbers of Preservatives: Sodium Bisulfate:_____ Methanol:_____" indicates a freezing option was employed, with or without the addition of water.

9/16/10:jad

1651

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SOP 1.3.2.2

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 480B SOP Section #: 1.3.2.2 Revision #: 15

SOP Title: Analysis of Volatile Organic Compounds in Aqueous and Medium/High Concentration Soil Samples by SW-846

Effective date: (QA fills in) 4-11-11

- Procedure prepared by: [signature] Date: 4.8.11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 4-11-11
- Reason for change: add additional DoD-QSM protocols and parameters, annual review
- This procedure meets the requirements of the following approved method references: SW-846 3RD Edition, Update III, 12/96, Method 8260B, Method 5030B, Method 5035; Dept. of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 480B: Analysis of Volatile Organic Compounds in Aqueous and Medium/High Concentration Soil Samples by SW-846

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Instrument Procedure 480B: Analysis of Volatile Organic Compounds in Aqueous and Medium/High Concentration Soil Samples by SW-846

1.0 Scope and Application

This is a general purpose method for the identification and simultaneous measurement of purgeable volatile organic compounds in a variety of aqueous matrices following SW-846 Method 8260B. It is also used to analyze medium to high level soil samples that have been extracted with methanol. The method is applicable to a wide range of organic compounds. It incorporates Method 5030B and Method 5035 (for medium/high concentration soil samples, extracted with methanol). Target compounds for this method, along with their associated internal standards and quantitation ions, are listed in Attachment 1. Note, however, that many of these compounds are not routinely analyzed.

The reporting limit is the low level calibration standard concentration. Reporting limits for this method are shown in Attachment 2.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

An inert gas (helium) is bubbled through a 5 mL or 25 mL aqueous sample spiked with internal standard and surrogate compounds. For medium/high level soil samples, an aliquot (typically 100 µL) of the methanol extract containing surrogates (See SOP 1.1.4.1) is added to 5 mL nitrogen-sparged reagent water. The sample is purged in a 40 mL VOA vial at ambient temperature, causing the purgeable volatile organic compounds to be transferred from the aqueous phase to the vapor phase. The vapor is swept onto a sorbent column where the purgeables are trapped. After purging is completed, the sorbent column is heated and back-flushed with the inert gas to desorb the purgeables onto a gas chromatograph (GC) wide-bore capillary column. The GC is temperature-programmed to separate the purgeables that are then detected with a mass spectrometer (MS). This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

Note: For Method 8260B, a heated purge is also allowed.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than

the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.**6** Reporting Units – µg/L for water and µg/Kg for soil

3.**7** A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.**8** SC DHEC – South Carolina Department of Health and Environmental Control

3.9 **DoD**-QSM – Department of Defense Quality Systems Manual

3.10 Marginal Exceedance – Beyond the LCS control limit but within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

3.11 Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.12 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Impurities in the purge gas or methanol, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. Gas lines from the gas tanks to the

instrument must be either stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. When potential interfering peaks are noted in laboratory method blanks, it may be necessary to reduce solvent contamination in the laboratory, purge the methanol used to prepare standard solutions, purge the DI water with helium or nitrogen, change the purge gas source, or regenerate the molecular sieve purge gas filter.

4.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples are stored in GC/MS VOA laboratory refrigerators; separate from laboratory standards, and they must be analyzed in a room in which the atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. Because methylene chloride will permeate PTFE tubing, all GC carrier gas lines and purge gas plumbing are to be constructed from stainless steel or copper tubing.

4.3 Contamination by carryover can occur whenever a sample is analyzed after a sample that contains high levels of organic compounds. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank or the next sample must be closely monitored to check for cross-contamination. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high purgeable levels, it may be necessary to clean the purge and trap apparatus. Do so by purging a 10-20% methanol solution, followed by baking the purge and trap apparatus and the analysis of a DI water blank to confirm that the system is free from contamination. The trap and other parts of the system are also subject to contamination; therefore, frequent bake out and purging of the entire system may be required.

4.4 Instrument Problems/Preventative Maintenance

Instrument problems may interfere with the analysis. If a low response is observed for the early eluting compounds such as the gases, replacement of the trap or septum may be necessary. In addition, adjustments to the purge flow may be necessary to achieve a desired response for these compounds. If such adjustments do not help, it may be necessary to check the fittings on the purge and trap device and on the column for leaks. This is done with a helium leak detector and certain software utility programs. Column maintenance or replacement may be necessary if peak tailing or broad chromatographic peaks are observed.

5.0 Safety

5.1 The toxicity and carcinogenicity of many chemicals used in this method have not been precisely determined. Each chemical should be treated as a potential health hazard. Exposure to these chemicals should be minimized. Preparation of calibration standards, blanks, and samples is performed in a fume hood to minimize risk.

5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2- dichloroethane, 1,1,2,2-tetrachloroethane, 1,1,2- trichloroethane, chloroform, 1,2-dibromoethane, trichloroethene, and vinyl chloride.

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff members are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Syringes

6.1.1 1 mL Hamilton syringe

6.1.2 500 µL Hamilton syringe

6.1.3 100 µL Hamilton syringe

6.1.4 50 µL Hamilton syringe

6.1.5 25 µL Hamilton syringe

6.2 Volumetric Flasks and Pipets

6.2.1 Assorted volumetric flasks ranging from 50 mL to 1000 mL

6.2.2 10 mL graduated pipette in 1/10 mL graduations

6.2.3 One mL mininert vials

6.3 Vials

6.3.1 40 mL screw-top, PTFE-lined, septum-sealed vials, with and without magnetic stirring bars

6.4 Analytical Column

6.4.1 Supelco SPB-624 60-m, 0.32mm ID with 1.8 um film thickness

6.4.2 Descriptions of alternative capillary columns are presented in EPA CLP SOW documents. Columns other than those listed may be used if equivalency is demonstrated. Specifications for equivalency are also presented in the SOW documents. The equivalency documentation must be maintained and made available during on-site audits

6.5 Purge and trap Autosampler System

6.5.1 Tekmar LSC

6.5.1.1 Tekmar, LSC 3000, LCS 3100 and Velocity XPT with glass frit bottom liquid sample purging vessel and Luerlock valve

6.5.1.2 The absorption trap must be at least 25 cm long and have an internal diameter of at least 0.105 inches (0.2667 cm).

6.5.1.3 The Supelco K (VOCARB 3000) trap consists of the following:

- Carbopack B, 10 cm
- Carboxen-1000, 6 cm
- Carboxen-1001, 1 cm

6.5.1.4 Alternate sorbent traps may be used if they meet the QC acceptance criteria. Performance documentation must be maintained and made available during an on-site audit.

6.5.2 Archon ALS

6.5.2.1 The Archon Model 5100, 4552 Purge and trap autosampler, interfaces directly to a Tekmar Purge and Trap Concentrators.

6.5.2.2 The autosampler is designed for soil samples and utilizes 40 mL VOA vials with low bleed Teflon septa.

6.5.2.3 The Archon ALS has the capacity of up to 51 vials.

6.5.3 Purge and Trap Analysis

6.5.3.1 After preparation, load each standard, blank, and sample onto the closed-system autosampler interfaced with the purge-and-trap apparatus.

6.5.3.2 Purge for 11 minutes at 40 ºC.

6.5.3.3 Heat the trap to 180 ^{o}C and desorb the trapped contents onto the GC column by back flushing the trap with inert gas for 4.0 (± 0.1) minutes.

6.5.3.4 Bake the trap for 7.0 (± 0.1) minutes at 220 to 260 ^{o}C.

6.6 Gas Chromatograph / Mass spectrometer

6.6.1 Hewlett Packard 5890 Series

6.6.2 Hewlett Packard 6890 Series

6.6.3 Agilent 6890N Series

6.6.4 Hewlett Packard 5972 Mass Selective Detector (MSD)

6.6.5 Hewlett Packard 5973 MSD

6.6.6 Hewlett Packard 6890MSD

6.6.7 Agilent Technologies 5975 MSD

6.7 HP/ Agilent GC conditions listed below:

- Column: 30m

- Carrier Gas: Helium
- GC mode: Capillary
- Injection Port Temperature: 230°C
- Interface Temperature: 230°C
- Initial Temperature: 40° C
- Final Temperature: 225° C
- Flow Rate: 35ml/minute

The listed column flow rate is approximate. The flow rate is adjusted to optimize the linear velocity of an un-retained compound (butane) through the column. The optimum linear velocity for a SPB-624 column is 30 - 45 cm/second.

6.8. Temperature Program

These temperature program conditions are provided as an example and may vary.

- HP/Agilent: 40 °C for 4.50 minute
- Ramp: 40 °C – 100 °C @ 20 °C/minute
- Ramp: 100 °C – 150 °C @ 25 °C/minute
- Ramp: 150 °C – 225 °C @ 33 °C/minute

6.9 Interface (GC to mass spectrometer)

6.9.1 Direct capillary interface at 225° C is used for Hewlett Packard MSDs.

6.10 Data system

6.10.1 The Hewlett Packard GC/MS systems utilize ChemServer software, for data acquisition.

6.10.2 For data processing, CompuChem uses the Hewlett Packard HP 9000 series 735 Unix Workstation employing Target3 and Envision software by Thru-Put Systems.

6.10.3 The reference library used is the NIST Library (NIST129K.1), G1033A version D.0500 purchased from Quantum Analytics Inc.

6.11 Data storage

Data are stored on the Target3 Unix Workstation. The Workstation is backed up incrementally on a daily basis.

6.12 Element DataSystem® LIMS computer software by Promium is used for sample management and report generation.

7.0 Reagents and Standards

Note: Detailed standard preparation information may be found in the Standard Preparation Logbook 11D(4), "GCMS VOA Standard Preparation Logbook", located in the Volatile Laboratory or in LIMS.

All standards are prepared in the Volatile Organic Laboratory. Stock Standards are stored separately from samples in Teflon- sealed screw-cap bottles with zero headspace at -10 to -20 ^{o}C in the freezer units in the instrument laboratory when not in use. Protect the standards from light. Standards for gases usually need to be replaced after one week or as recommended by the manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases must be monitored and fresh standards prepared if a 20% drift is experienced. These standards need to be replaced after six months or as recommended by the manufacturer, unless the acceptability of the standard can be documented. 2-chloroethyl vinyl ether (CEVE) and styrene may have to be prepared more frequently. Secondary dilutions of Stock Standards must be checked frequently for signs of degradation or evaporation, especially just prior to their use in preparing the working calibration standards. Working standards must be prepared just prior to analysis unless they are to be purged by an autosampler. When an autosampler is used, the standards may be kept up to 12 hours in purge vessels connected via the autosampler to the purge and trap device.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: **All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.** All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration

7.1 Reagent Water-All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methanol (B&J Scientific, purge and trap grade)

7.3 Tuning Standard

7.3.1 4-Bromofluorobenzene - Standard ID# 7008 at 25 µg/mL. 2 µL yielding 50 ηg on column, are injected onto the column every 12 hours.

7.3.1.1 Prepare the standard by adding 50 µL Restek VOA Tuning Mix 5000 µg/mL) to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.4.1.2 Prepare this standard monthly.

7.4 Calibration Standards

7.4.1 For the initial calibration, the internal standard solution is added automatically by the Archon Purge and Trap Autosampler. For all subsequent analyses, both the internal standard and the surrogate solutions are added automatically by the Archon autosampler.

Standard Preparation Table 2

Std. ID TCL4-High	005 µg/L	010 µg/L	020 µg/L	050* µg/L	100 µg/L	200 µg/L
8260 I.S.	5.0 µL	5.0 µL	5.0 µL	5.0 µL	5.0 µL	5.0 µL
8260 S.S.	1.0 µL	2.0 µL	5.0 µL	10.0 µL	15.0 µL	20.0 µL
TCL4-1&2	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL
TCL4-gases	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL
TCL4-ketones	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL
TCL4-AppIX	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL

* Continuing Calibration level

7.4.1.1 To prepare the standards at the concentrations shown in the column headers of the preceding table, add the µL amount of standard shown to a 100 mL volumetric flask containing nitrogen sparged DI water, then bring up to volume.

Alternatively, the standards may be prepared at the above concentrations by diluting the 200 µg/L standard directly into the purge and trap impingers. Dilute as follows:

- For a 100 µg/L standard, add 2.5 mL to 5 mL DI water;
- For a 50 µg/L standard, add 1.25 mL to 5 mL DI water;
- For a 20 µg/L standard, add 0.50 mL to 5 mL DI water;
- For a 10 µg/L standard, add 0.25 mL to 5 mL DI water;
- For a 5 µg/L standard, add 0.125 mL to 5 mL DI water.

For a 25 mL purge analysis, increase both volumes by a factor of 5 when preparing standards by making dilutions.

Note: For samples submitted to meet the regulatory requirements of the State of South Carolina, this initial calibration curve must include a standard at 2 µg/L.

7.4.1.2 The concentration of the compounds in the TCL4-1 & 2 High standard is 500 µg/mL. See Attachment 5 for the composition of this standard.

7.4.1.2.1 Prepare the standard by adding 1.25mL Restek 502.2 VOA 2000 MegaMix (2000 µg/mL) to an amount of purge and trap grade methanol in a 5 mL volumetric flask and bring to volume.

7.4.1.2.2 Prepare this standard every three months.

7.4.1.3 The concentration of the compounds in the TCL4-gases High standard is 500 µg/mL. See Attachment 5 for the composition of this standard.

7.4.1.3.1 Prepare each standard by adding 1.25 mL Restek 502.2 Calibration Mix #1 (2000 µg/mL) to an amount of purge and trap grade methanol in a 5 mL volumetric flask and bring to volume.

7.4.1.3.2 The method states that this standard usually needs to be replaced weekly, unless the standard manufacturer recommends otherwise, or unless the acceptability of the standard can be documented. This standard has generally proven to be more stable in the laboratory. The gas standard can be used for longer than a week if the gases in the continuing calibration (CCV) standard meet the CCV requirements when compared to the initial calibration standards that contain a gas standard

that has been prepared within a one week holding time. Prepare this standard monthly, or more frequently as need dictates, or when degradation is evident rendering the standard unacceptable.

7.4.1.4 The concentrations of the compounds in the TCL4-ketones standard are 500 µg/mL, 1250 µg/mL, or 5000 µg/mL. See Attachment 5 for the composition of this standard.

7.4.1.4.1 Prepare the standard by transferring AccuStandard custom VOA Mix #2 to a mininert vial.

7.4.1.4.2 Replenish this standard as needed and replace every three months.

7.4.1.5 The concentrations of the compounds in the TCL4-AppIX standard are 500 µg/mL, 5000 µg/mL, or 25000 µg/mL. See Attachment 5 for the composition of this standard.

7.4.1.5.1 Prepare the standard by transferring AccuStandard custom VOA Mix #1 to a mininert vial.

7.4.1.5.2 Replenish this standard as needed and replace every three months.

7.5 Initial Calibration Verification

7.5.1 The initial calibration curve must be verified using a standard from an independent source. The laboratory purchases the initial calibration verification (ICV) standard from a different vendor than the one used for the calibration standards. This is prepared in the same manner as described above.

7.5.2 The ICV contains the full list of target analytes at the same concentration as the continuing calibration verification (CCV) standard.

7.6 Internal Standards

Compounds in 8260 I.S. (Internal standard) at a concentration of 50 µg/ml (250 ug/ml for the Archon)

- Fluorobenzene
- D5-chlorobenzene

- D4-1,4-dichlorobenzene

7.6.1 Prepare the internal standard spiking solution by adding 0.2 mL Restek 8260A ampulated internal standard mix (2500 µg/L to an amount of purge and trap grade methanol in a 10 ml volumetric flask and bring to volume.

7.6.2 Prepare this standard every three months.

7.7 Surrogate Standard

Compounds in 8260 S.S. (Surrogate Standard) at a concentration of 50 µg/mL (250 µg/mL for the Archon)

- Dibromofluoromethane
- D4-1,2-dichloroethane
- D8-toluene
- 4-bromofluorobenzene

7.7.1 Prepare the surrogate spiking solution by adding 0.2 mL Restek 8260A ampulated surrogate mix (2500 µg/mLl) to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.7.2 Prepare this standard every three months.

7.8 Spike Standard

Compounds in 8260 Spiking Mixture (1001C) at a concentration of 25 µg/mL

- 1,1-dichloroethene
- Trichloroethene
- Benzene
- Toluene
- Chlorobenzene

Note: The spiking cocktail is project dependent. This spiking mixture can also be used as a Laboratory Control Sample (LCS) spike. Some projects may require full analyte spike, and in that case, the standard used for the full analyte spike LCS is the ICV. For some programs, the CCV may be used in the place of the LCS.

7.8.1 Prepare this standard by adding 100 µL Restek VOA Matrix Spike Mix to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.8.2 Prepare this standard every six months.

7.9 Standard Storage

7.9.1 Store the stock standards in Teflon- sealed screw-cap bottles with zero headspace at -10 °C to -20 °C. Protect the standards from light. Standards for gases usually need to be replaced after one week or as recommended by the manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases should be monitored and fresh standards prepared if a 20% drift is experienced. These standards need to be replaced after six months or as recommended by the manufacturer, unless the acceptability of the standard can be documented. CEVE and styrene may have to be prepared more frequently.

7.9.2 Store secondary dilution standards in Teflon®-sealed screw-cap bottles with minimal headspace at -10 °C to -20 °C. Protect the standards from light. The secondary dilution standards must be checked frequently for signs of degradation or evaporation, especially just prior to their use in preparing the working calibration standards.

7.9.3 Aqueous working standards must be prepared just prior to analysis unless they are to be purged by an autosampler. When an autosampler is used, the standards may be kept up to 12 hours in purge vessels connected via the autosampler to the purge and trap device. If premixed certified solutions are used store according to manufacturer's documented holding time and storage temperature recommendations.

7.9.4 Purgeable standards are stored in GC/MS VOA Freezer #1, separate from other standards and samples.

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples are preserved and stored according to the tables in Sample Control SOPs 4.1, “Receiving Samples” and 4.6, “Storing Samples.” Sample holding times are also listed.

8.1.1 Note that if 2-chloroethyl vinyl ether (CEVE) is a target compound of interest for the project, an unpreserved sample must be analyzed within 7 days of collection.

8.2 All samples must be analyzed within 14 days of collection.

8.3 Prior to analysis, all samples must be stored under refrigeration at 2 – 6 ^{o}C in the reach-in storage unit in the laboratory. After analysis, samples are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 All field and QC samples are spiked with the surrogate standard. Surrogates are used to assess the efficiency of the analytical system.

9.1.2 Surrogate compounds must meet the following percent recovery criteria.

Surrogate Compound	**Aqueous and Med./High Conc. Soil**	**Aqueous**
	5 mL purge	**25 mL purge**
Dibromofluorobenzene	66-128; 71-141	65-150
1,2-Dichloroethane-d_4	55-147; 70-139	59-150
Toluene-d_8	50-150; 72-123	61-145
4-bromofluorobenzene	70-132; 65-131	63-143

9.1.2.1 The same surrogates and recovery criteria are to be used for samples submitted to meet the regulatory requirements of the State of South Carolina.

The following table contains the surrogate recovery limits required by the **DoD**-QSM.

Surrogate Compound	Aqueous	Solid
Dibromofluorobenzene	85-115	N/A
1,2-Dichloroethane-d_4	70-120	N/A
Toluene-d_8	85-120	85-115
4-Bromofluorobenzene	75-120	85-120

9.1.3 Samples with surrogate recovery failures must be reanalyzed to confirm a matrix effect. Surrogate recovery failures in method blanks and LCS require reanalysis of the entire batch. Similar surrogate failures in the MS/MSD and original sample confirm a matrix effect and do not require reanalysis.

9.2 Internal Standards

9.2.1 The integrated areas of the quantitation ions of the internal standards are monitored in continuing calibration verification checks, samples, and QC for a change in retention time and response or sensitivity. These should remain reasonably constant over time.

9.2.1.1 Internal standard retention time and area responses must be assessed in each continuing calibration verification standard by comparison to the corresponding internal standard in the most recent initial calibration mid-point standard. Internal standard responses in samples and QC are compared to the most recent continuing calibration verification.

9.2.2 The area responses of the internal standards must be within 50-200% difference of the area responses compared to.

9.2.3 The retention time shift for the internal standards must be less than 30 seconds.

9.2.4 If any of these criteria cannot be met, the analytical system must be checked for malfunctions and corrections made. Re-analysis of any affected sample is required.

9.3 Method/Instrument Blanks

9.3.1 Before any samples are analyzed, it must be demonstrated through a laboratory reagent blank that the system is free of contamination that would prevent the determination of any analyte of concern. Sources of background contamination are glassware, purge gas, sorbents, and equipment. Background contamination must be reduced to an acceptable level before proceeding with the next analysis. In general, background contamination from method analytes should be below the reporting limit.

9.3.2 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, and continuing calibration verification acceptance criteria.

9.3.3 A method blank is analyzed with each batch of up to 20 samples processed as a group within a 12-hour tune. If more than 20 samples are analyzed in a tune batch, a second method blank is required. Method blanks must be analyzed immediately following a valid continuing calibration verification analysis

9.3.4 The concentration of the target compounds in the blank must be less than the reporting limit for each target compound, with the exception of the common laboratory contaminants acetone, 2-butanone, and methylene chloride, which must be <2X the reporting limit..

9.3.4.1 The **DoD**-QSM requires that the target compounds in the blank must be at a concentration of <½ the reporting limit and < the reporting limit for lab contaminants.

9.3.4.2 The SC DHEC requires that all compounds in the blank be less that the reporting limits except for the **common laboratory contaminants**, which must be <2X the reporting limit.

9.3.5 All samples processed within the same 12-hour tune with a method blank that does not meet the blank technical acceptance criteria must be reanalyzed. The chromatographic system must be inspected for malfunctions, and corrections must be made as required before more samples are analyzed.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS) is prepared and analyzed with each tune batch of up to 20 samples. The LCS and matrix spikes are spiked with the same target analytes. The LCS is spiked at the same concentration as the matrix spike. The LCS is spiked with all compounds of interest in the project.

9.4.2 The percent recovery criteria, developed from in-house statistical data, for the analytes in the full LCS are listed in Attachment 3.

9.4.2.1 The LCS control limits required by the DOD-QSM are listed in Attachment 6 along with the marginal exceedances.

9.4.2.2 The following table presents the DOD-QSM and 2003 NELAC Standards allowed number of marginal exceedances governed by the number of compounds spiked into the LCS.

Number of analytes in the LCS	Allowed number of Marginal Exceedances
> 90	5
71-90	4
51-70	3
31-50	2
11-30	1
< 11	0

The LCS fails if the more than the allowed number of marginal exceedances occur or if a spike recovery is outside of the marginal exceedance limits. The SC DHEC does not allow for marginal exceedances

9.4.3. Gases and known poor purging compounds

- gases: bromomethane
  chloromethane
  chloroethane
  vinyl chloride
  dichlorodifluoromethane
  trichlorofluoromethane
- acetone
- 2-butanone
- carbon disulfide
- crotonaldehyde
- 1,2-dibromo-3-chloropropane
- 1,4-dioxane
- isobutyl alcohol
- 2-hexanone
- 4-methyl-2-pentanone
- vinyl acetate

9.4.4 For SC DHEC, an expanded subset of analytes, representative of the compounds being reported, is employed and all analytes must have recovery limits within 70-130%. The analytes are:

- vinyl chloride
- 1,1-dichloroethene
- methylene chloride
- 1,1-dichloroethane
- cis-1,2-dichloroethene

- 2-butanone
- carbon tetrachloride
- benzene
- trichloroethene
- 1,2-dichloropropane
- bromodichloromethane
- tetrachloroethene
- chlorobenzene
- ethylbenzene
- styrene
- bromoform
- 1,4-dichlorobenzene

9.4.5 When the LCS fails to meet the acceptance criteria, the entire batch associated with it must be re-prepared and reanalyzed.

9.5 Matrix Spikes

9.5.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared and analyzed with every SDG.

9.5.2 Matrix spikes have the following advisory recovery criteria:

Spike Compound	Aqueous & med./high conc. soil % Recovery
1,1-dichloroethene	61-145; 59-172
Trichloroethene	71-120; 62-137
Benzene	76-127; 66-142
Toluene	76-125; 59-132
Chlorobenzene	75-130; 78-122
All others	50-150

9.5.3 Matrix spikes have the following advisory relative percent difference (RPD) criteria as shown in the following table.

Spike Compound	Aqueous & med./high conc. soil % RPD

Spike Compound	Aqueous & med./high conc. soil % RPD
1,1-dichloroethene	14; 22
Trichloroethene	14; 24
Benzene	11; 21
Toluene	13; 21
Chlorobenzene	13; 21
All others	25

9.5.4 Most spike compounds should meet these criteria. If the criteria are not met in the MS/MSD but are met in the LCS, the results may be reported with the failures attributed to the matrix of the sample. If the LCS does not meet criteria, then all will have to be repeated as discussed above.

9.5.5 To meet the requirements of the **DoD**-QSM, the duplicate matrix spikes should meet the LCS control limits listed in Attachment 6. The RPD between the duplicate matrix spikes should be ≤ 30%.

9.5.5.1 If the duplicate matrix spikes fail **DoD**-QSM acceptance criteria, contact the client for guidance.

9.5.5.2 If the original sample results are associated with failing duplicate matrix spikes, qualify the results in the narrative as estimated concentrations. Refer to the **DoD**-QSM "J" flag.

9.6 Initial Calibration Verification

9.6.1 A second source initial calibration verification (ICV) standard is run after the initial calibration standards have met criteria.

9.6.2 The ICV must be within 20% of its expected value for each target analyte and surrogate or within 40% for the poor purgers and the gases. Sporadic failure of up to three target compounds is allowed but they must not exceed 40% of their expected value; gases and poor purgers are listed above.

9.6.2.1 To meet the requirements for the **DoD**-QSM, the ICV must be ≤ 25% of its expected value for each target analyte.

9.6.3 If the ICV fails to meet the criteria in **Sections** 9.6.2 or 9.6.2.1, take corrective action and reanalyze the standard. If the ICV fails again, repeat the initial calibration.

9.7 MDL Studies

9.7.1 On an annual basis and after major maintenance, a method detection limit (MDL) study is performed on at least one instrument per method and matrix. When multiple instruments are used, individual instrument MDL studies may be replaced by the analysis of an MDL check sample. The MDL check sample must be analyzed on all instruments, to demonstrate equivalent sensitivity.

9.7.1.1 The **DoD**-QSM requires that the MDL check sample be prepared at about 2x the MDL and is analyzed on a quarterly basis for each matrix. A response must be detected in the 2x MDL check sample. Additionally, qualifying ions of 50% or higher must also be present. For more information on MDL studies, refer to QC SOP 13.11, "Performing Annual Method Detection Limit (MDL) Studies."

9.8 Contingency

9.8.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.8.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.8.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.8.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 <u>Calibration **and** Standardization</u>

10.1 BFB Tuning

10.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ηg of BFB (2 µL STD ID#7008) into the GC using a 10 µL Hamilton syringe. BFB may be analyzed simultaneously with a continuing calibration verification standard as long as all QC criteria are met.

10.1.2 The peak selection criteria for BFB analysis are as follows (in order of performance):

10.1.2.1 Average one scan prior to the apex of the BFB peak to one scan after the apex, subtracting a single background scan prior to the peak, but no more than 20 scans prior to the elution of BFB. Also, do not subtract part of the BFB peak.

10.1.2.2 Choose the apex of the BFB peak only and include background subtraction.

Note: Background subtraction is performed to eliminate interference and when performed, the subtracted scan must be no more than 20 scans prior to the elution of the BFB and no scans within the BFB peak may be subtracted.

10.1.2.3 Choose a single scan or a range of scans within the BFB peak and include background subtraction.

10.1.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given in the following table.

BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15-40% of m/z 95
75	30-60% of m/z 95
95	Base Peak; 100% relative abundance
96	5-9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95
175	5-9% of m/z 174
176	>95% but less than 101% of m/z 174

Mass	Ion Abundance Criteria
177	5-9% of m/z 176

10.1.4 BFB technical acceptance criteria must be met before any standards, samples, or required blanks are analyzed. Tune criteria must be met 12 hours. GC/MS tuning and Mass Calibration forms must be printed and attached to the instrument run log page for each tune. The relative abundance for each ion is calculated to two decimal places.

10.1.5 If BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, or take other corrective action to achieve the technical acceptance criteria.

10.2 Initial Calibration

10.2.1 Prior to the analysis of samples and required blanks, and after the instrument performance check solution (BFB) criteria have been met, each GC/MS system must be calibrated at six concentrations to demonstrate instrument sensitivity and the linearity of responses for the purgeable target compounds.

10.2.2 Prepare standards according to the Initial Calibration Standard Preparation Table 2 in Section 7.4. All initial calibration standards must be analyzed at the concentration levels and frequency described in this SOP on a GC/MS system that meets the BFB technical acceptance criteria.

10.2.3 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard. Relative response factors (RRF) are calculated for each compound.

10.2.4 Minimum relative response factors for the System Performance Check Compounds (SPCC) must be met and are listed in the following table.

Relative Response Factor Criteria for SPCCs

Volatile Compound	**Minimum RRF**
Chloromethane	0.10
1,1-dichloroethane	0.10
Chlorobenzene	0.30

Volatile Compound	**Minimum RRF**
Bromoform	0.10
1,1,2,2-tetrachloroethane	0.30

10.2.5 The following Calibration Check Compounds (CCC) have maximum % RSD criteria of ≤ 30%.

- 1,1-dichloroethene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene
- vinyl chloride

10.2.5.1 The remaining compounds must have an RSD of ≤ 15%. If the % RSD is 15% or less, the average relative response factor may be used for quantitation. If the % RSD is greater than 15% then an alternate method for quantitation, such as a linear calibration using least squares regression or quadratic fit, may be used. When one of these options is used, the line must not be forced through the origin. The SC DHEC does not allow the use of a quadratic regression for any methods that have previously shown linearity.

If the linear or quadratic regression fit is used, the correlation coefficient must be ≥ 0.990. These alternate methods of quantitation are available in the ThruPut system.

Note: The **DoD**-QSM requires that the linear least squares regression correlation coefficient must be ≥ 0.995 and does not allow the used of quadratic fit.

Note: If quadratic regression is used the compound must historically exhibit a nonlinear response.

10.2.5.2 If the initial calibration does not meet the criteria above, corrective action is necessary.

- Check the instrument operating conditions and perform maintenance as necessary. It may be necessary to clean the ion source, perform column maintenance, change the

column, service the purge and trap device, or take other corrective action to achieve the technical acceptance criteria.

- Compare responses for the analyte in each of the standard levels to verify that a single standard analysis is not presenting results significantly higher or lower then the other standard analyses, as this would indicate that the standard solution was prepared in error. If that is the case, re-prepare and reanalyze the standard.

10.2.6 The initial calibration may still be acceptable when some analytes exceed the 15% RSD criteria, if the following conditions are met and allowed by the client:

- The mean of all %RSD values for the analytes (grand mean) is less than or equal to 15%.

- All analytes in the calibration standard must be included in the calculation.

- Non-CCC target compounds have a warning limit of 50% RSD and an action limit of 90% RSD when the "grand mean" approach is used. These limits have been inserted as default values into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD result in rejection of data.

10.2.6.1 A summary of the initial calibration data and/or a list of the analytes not meeting the 15% RSD criteria with their actual %RSD must be included as a deliverable to our client. If the conditions in 10.2.6 are met, then the average relative response factor may be used to determine the concentration of analytes in samples.

Note: For samples submitted to meet the regulatory requirements of the State of South Carolina and the **DoD**-QSM, the grand mean option is not allowed.

10.2.7 The initial calibration verification must be analyzed after each initial calibration and must meet the acceptance criteria. If the ICV fails, then a new initial calibration curve must be generated.

10.3 Continuing Calibration Verification

10.3.1 Before the analysis of samples and blanks, but after BFB and initial calibration acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a continuing calibration verification standard. This standard must contain all purgeable target analytes and surrogate compounds. It is used to ensure that the instrument meets the sensitivity and linearity requirements of the method throughout the analytical sequence.

10.3.2 A check of the calibration curve must be performed once every 12 hours, beginning with the injection of BFB. A percent difference of the response for each compound compared to the mean relative response factor from the initial calibration is calculated when performing the average response factor model.

10.3.3 The calculated percent difference must be less than or equal to 20% (%D) for the CCCs listed above in Section 10.2.5. Minimum response factor criteria for the continuing calibration verification standard are also shown in Section 10.2.4.

10.3.3.1 If a regression fit model was used for analytes in the initial calibration, the continuing calibration verification is performed using percent drift (difference) for the CCCs.

10.3.4 As indicated for the initial calibration acceptance criteria, for the continuing calibration verification, the remaining target analytes (non-CCC compounds) do not have defined % difference criteria. We have established a warning limit of 50%D and an action limit of 90%D. These values have been inserted as defaults into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD result in rejection of data.

10.3.4.1 For samples submitted to meet the regulatory requirements of the State of South Carolina, all non-CCC target analytes must be within 30% acceptance criteria, with the exception of the poor purgers listed in Section 9.4.3. The poor purgers should fall within 40% acceptance criteria.

10.3.5 If continuing calibration verification acceptance criteria cannot be met after inspection and normal maintenance, a new initial calibration will have to be performed.

Note: Method 8260B indicates that if the CCCs are not required analytes, then all required analytes, must meet the 20% difference criterion. Our typical analysis includes all of the CCCs.

11.0 Procedure

Documentation must follow the requirements in QC SOP 13.6: "Proper Documentation Procedures". All injections must be recorded on the instrument run log (Attachment 4) along with the date, time (use a 24 hour clock), the volume injected, operator ID, and any comments relevant to the injection.

All standards, blanks, samples and other required runs must be injected within exactly 12 hours from the time of the injection of BFB.

11.1 Instrument Software Conventions

11.1.1 Quantitation method: Average response factor

11.1.2 File naming conventions:

11.1.2.1 Name calibration standards and instrument blanks as follows:

YMDDZZZ-XXX#

where: Y = Year

M = Month (A=Jan. B=Feb…)

DD = two digit day

ZZZ = three digit sequence number

XXX = QC Type (Tun=BFB, ICV= Initial calibration, CCV=continuing cal)

= numerical order

11.1.2.2 Name sample analyses as follows:

CCN-##R# or CCN##D#

where: CCN = work order number designated for the sample by LIMs

= the sample number (typically 01-20)

R# = indicating reanalysis of the sample (R1, R2…)

D# = indicating diluted analysis of the sample (D1, D2…)

11.2 Analytical Sequence

11.2.1 Order of analysis for the instrument calibration

- BFB (tune)
- initial calibration
- initial calibration verification

11.2.2 Order of analysis for the twelve-hour tune

- BFB
- continuing calibration verification
- instrument blank
- laboratory control sample
- samples

11.2.3 In some cases, if tune time remains after the initial calibration standards have been run, samples may be analyzed as long as they are preceded by an acceptable instrument blank and LCS.

11.2.4 All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.3 Preparations

11.3.1 Standards

11.3.1.1 Load the standard solutions onto the purge and trap apparatus. Purge for 11 minutes at ambient temperature, or 40° C for heated purge, and desorb for 4 minutes, analyzing all target compounds.

11.3.2 Method/Instrument Blanks

11.3.2.1 Method/instrument blanks are prepared by filling one 25 mL or 5 mL gastight syringe with DI water and spiking with internal standard solution and surrogate solution.

11.3.3 Laboratory Control Sample spikes

11.3.3.1 Laboratory control samples (LCS) are prepared at 5.0 µg/L, with 5 µg/L of surrogates for 25 mL samples and at 50 µg/L, with 50 µg/L of surrogates for 5 mL samples.

11.3.3.2 For certain projects and programs, a full list spike is required.

11.3.4 Samples

11.3.4.1 Liquid samples are collected with zero headspace and provided to the laboratory in 40 mL or 60 mL screw-cap vials with Teflon-lined silicon septa. To prepare liquid samples for analysis, pour the sample into a 5 mL or 25 mL Hamilton gastight syringe. Replace the plunger and adjust the volume to 5 mL or 25 mL. Spike each 25 mL sample with 2.5 µL of internal standard and 2.5 µL of surrogate solutions through the bore of the syringe. Spike each 5 mL sample with 5 µL of internal standard and 5 µL of surrogate solutions. Inject syringe contents into a 40 mL vial and place on the autosampler for analysis.

11.2.4.1.1 Take the pH of each water sample. After the sample aliquot is taken, add a couple of drops to pH paper. Do not add pH paper to sample vials. Record the pH of each sample on the batch sheet and GC/MS run log. No pH adjustment is to be performed.

11.3.4.2 Methanol extracts of medium/high concentration soil samples are contained in an autosampler vial capped with a Teflon-faced septum. The vial contains 1 mL of the methanol extract that also contains surrogates. A 5 mL Hamilton gastight syringe is filled with DI water and adjusted to 4.9 mL. The plunger is pulled back to 5.0 mL to allow for the addition of the methanol extract and internal standard solution. Add 100 µL of the methanol extract and 5.0 µL of internal standard solution to the syringe. Based on prior or screening results, if less than 100 µL of the methanol extract is required in order to get target analytes within the initial calibration range, add an additional amount of

methanol to total 100 µL. Inject the contents of the syringe into a 40 mL vial and place on the autosampler for analysis.

11.3.4.3 For further details, see Sample Preparation Procedure –238, "**Preparation of Soil/Sediment/Sludge/Non-aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap by SW-846 Methods 5035A and 3585, and EPA CLP SOW OLM04.3**", SOP 1.1.4.1**.**

11.3.6 Matrix Spikes

11.3.6.1 For matrix spikes, in addition to spiking internal standard solution and surrogate solution, also add 5.0 µL of 8260B spike solution. For certain projects, a full target list matrix spikes are required. For medium/high concentration soil samples, the spiking solution is added at the time of the methanol extraction.

11.4 Analysis

11.4.1 Purge the sample for 11.0 (± 0.1) minutes.

11.4.2 After purging, the purge and trap apparatus will desorb onto the GC column by elevating the trap temperature to 260 °C and back-flushing the trap with helium for 4 minutes at 20 to 60 mL/minute.

11.4.3 After desorbing, the trap is reconditioned by baking at 260 °C for at least 7 minutes. When the trap has finished baking and is cool, it is ready for the next sample to be purged.

11.4.4 In each analytical run, all analytes must fall below the maximum calibration range established by the highest standard in the initial calibration. If an analyte is present at a concentration higher than the highest initial calibration standard, it must be reanalyzed at a lesser amount or dilution. A valid dilution is one in which the compound in question falls within the mid and high point calibration standard concentration.

11.5 Target Compound Identification

11.5.1 Target compounds are identified in the samples by analyzing standards under the same condition. Resultant mass spectra are compared to established library spectra and GC retention times are compared to retention times from the most recent continuing calibration standard. The

mass spectrum of the sample compound and a laboratory library-generated spectrum must match according to the following criteria.

11.5.1.1 All ions present in the library mass spectrum at a relative intensity >10% must be present in the sample spectrum.

11.5.1.2 The relative intensities of ions specified above must agree within ± 20% between the library and sample spectra.

11.5.1.3 Ions >10% in the sample spectrum but not present in the library spectrum must be considered and accounted for. (These ions may be a result of co-eluting/closely-eluting compounds.)

11.5.2 If a compound analyzed by GC/MS techniques cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the laboratory will report that identification.

11.5.3 Non-target compounds (tentatively identified compounds or TICs) are identified by comparing the mass spectra from the TICs to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library.

11.6 Qualitative Analysis of Non-Target Compounds

11.6.1 Tentatively identify all compounds in the sample that have not already been identified as target compounds, or that are not surrogates, internal standards or semivolatile target compounds, by performing a computer-generated library search using the NIST 129K.1 mass spectral library (See Section 6.6.2). The library search must not be normalized.

11.6.2 Up to 30 tentatively identified compounds (TICs), including alkanes, of the greatest concentration are reported for each sample. The number of TICs required may vary, depending on project requirements.

11.6.3 Rules for making tentative identification:

11.6.3.1 TICs receiving a library search match of 85% or higher are considered a “probable match”. Report the compound identified.

11.6.3.2 For TICs receiving more than one library search match of 85% or higher, report the compound with the highest percent match.

11.6.3.3 For TICs receiving two or more library search matches of 85% or higher with the same percent match, report the first compound.

11.6.3.4 For TICs that are isomers receiving library search matches of 85% or higher, report the compound with the highest percent match.

Note: If in the opinion the experienced analyst/data reviewer there is sound technical evidence not to identify the compound as specified in sections 11.6.3.1 to 11.6.3.4 the justification must be documented in the narrative.

11.6.3.5 TICs receiving a library search match of < 85% are given a tentative identification by the analyst/data reviewer, if possible. If no identification can be made the TIC is reported as "unknown". If possible the unknown is further identified as part of a class of compounds (for example "unknown aromatic").

11.7 Quantitation

11.7.1 A relative response factor is established for each target and surrogate compound during the initial and continuing calibration verification procedures. Quantitation of target analytes is based on the mean relative response factor from the initial calibration curve.

11.7.2 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative.

11.7.3 TICs are quantified by comparing the mass spectral response from the reconstructed ion chromatogram (RIC) for the TIC peaks to the mass spectral response for a peak produced by the nearest internal standard compound. A response factor of 1 is assumed.

12.0 Data Analysis and Calculations

Calculations must be consistent with the QC SOP 13.4: "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{\left(Value\ 1\ +\ Value\ 2\right)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value\ -\ \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax\ x\ C(is)}{A(is)\ x\ Cx}$$

where:
- Ax = Area of the characteristic ion (EICP) for the compound to be measured
- A(is) = Area of the characteristic ion (EICP) for the specific internal standard
- C(is) = Concentration of the internal standard (in µg/L)
- Cx = Concentration of the compound to be measured

12.8 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where:
- y = Instrument response (peak area)
- a = Slope of the line (coefficient of x)
- x = Concentration of the calibration standard
- b = The intercept

12.9 Concentration

12.9.1 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard.

12.9.2 Concentration of aqueous samples by GC/MS analysis using relative response factor:

$$\mu g / L = \frac{(Ax)(Is)(Df)}{(Ais)(\overline{RRF})(Vo)}$$

where:
- Ax = area of the characteristic ion from the EICP for the compound to be measured
- Ais = area of the characteristic ion for the EICP for the internal standard
- Is = amount of internal standard added (ng)
- $\overline{RRF}$ = mean relative response factor from initial calibration standards
- Vo = volume of water purged in milliliters
- Df = dilution factor. If no dilution, Df = 1.0

12.9.3 Concentration of medium level soil samples <u>(dry weight basis)</u> by GC/MS

$$\mu g / kg = \frac{(Ax)(Is)(Df)(Vt)(1000)}{(Ais)(\overline{RRF})(Ws)(Va)(D)}$$

where: Ax, Ais, Is, $\overline{RRF}$ are the same as given for water

Df = Dilution factor which, for medium/high concentration soil extract is defined by the following formula:

$$\frac{\mu L \text{ concentrated extract used for dilution} + \mu L \text{ clean solvent}}{\mu L \text{ concentrated extract used for dilution}}$$

Vt = Total volume of methanol extract, in mL

Note: This is typically 10 mL or 5 mL even though only 1 mL is transferred to the autosampler vial.

Ws = Weight of soil/sediment sample extracted, in grams (g)
Va = Volume of the aliquot of methanol extract

Note: Typically this is 100 µL but can be the volume of sample extract (not including the methanol added to equal 100 µL) in µL added to DI water.

Ws = weight of sample extracted, in grams

D (dry weight) = $\frac{100 - \%\text{ moisture}}{100}$

12.9.4 Concentration of aqueous and soil samples by GC/MS using quadratic (second order) fit in Target:

$$y = [n]\left[b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)\right]$$

where: b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
x = area of analyte/area of Internal Standard
n = amount of Internal Standard
y = concentration in ng on column
Rsp = area of analyte/area of Internal Standard

Example: Area of acetone = 35659

Area of IS = 613275
b = -0.0909161
m^1 = 9.605304
m^2 = 7.132688
ηg of IS = 250
response = 35659/613275 = 0.058145

Amount in ηg on column =

$$(250\,\eta g)[-0.0909161 + 9.605304 \;x\; 0.058145 + 7.132688 \;x\; 0.058145\;\text{^}2] = 122.9\eta g$$

Concentration (water) $\mu g / L = \dfrac{(122.9\eta g)(Df)}{(Vo)}$

Concentration (soil) $\mu g / Kg = \dfrac{122.9\eta g}{(Ws)(D)}$

12.9.5 Concentration of aqueous and soil samples by GC/MS using linear regression analysis:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$

$$C_s = \frac{\left[\dfrac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

where:
As = Area of the target analyte peak in the sample
Ais = Area of the internal standard peak
Cs = Concentration of the target analyte in the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (coefficient of Cs)
b = The intercept

Concentration (water) $\mu g / L = \dfrac{(Cs)(Df)}{(Vo)}$

Concentration (soil) $\mu g / Kg = \dfrac{Cs}{(Ws)(D)}$

12.9.6 Tentatively Identified Compound (TIC) Estimation

$$TIC\ Amount\ (water)\ \mu g/L = \frac{(Area\,TIC)\ \text{x}\ Amount\ (Std)(Df)}{Area(IS)\ x\ 1(RF)\ \text{x}\ (Vo)}$$

$$TIC\ Amount\ (soil)\ \mu g/Kg = \frac{(Area\,TIC)\ \text{x}\ Amount\ (Std)(Df)(Vt)}{Area\ (IS)\ x\ 1(RF)\ \text{x}\ (Vi)(Ws)(D)}$$

where:

Area (TIC)	=	area response from RIC for non-target compound
Amount (Std)	=	amount of internal standard added to the sample, in µg/L.
Area (IS)	=	area response of the nearest internal standard in the reconstructed ion chromatogram
1(RF)	=	assumed response factor of 1

12.10 Calculating Dilutions

12.10.1 The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters of water purged (i.e., Vo above) to the number of milliliters of the original water sample used for purging. For example, if 12.5 mL of sample is diluted to 25.0 mL with DI water and purged, Df = 25.0 mL/12.5 mL = 2.0. If no dilution is performed, Df = 1.0.

If a methanol extract contains an analyte that exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. Example: A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 8260B, Methods 5030B and 5035

16.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 QC SOP 13.6: "Proper Documentation Procedures"

16.5 QC SOP 13.4: "Numerical Data Reduction"

16.6 NELAC Standards, effective July 2003, plus revisions

16.7 QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.8 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **10**/**25**/**2010**

16.9 CompuChem Quality Manual, Revision 13, November 17, 2009

16.10 Sample Control SOP 4.1, "Receiving Samples"

16.11 Sample Control SOP 4.6, "Storing Samples"

16.12 QC SOP 13.11, "Performing Annual Method Detection Limit (MDL) Studies"

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 – Target Analyte List

17.2 Attachment 2 – Target Analyte Reporting Limits

17.3 Attachment 3 – In-house Statistical Control Limits

17.4 Attachment 4 – **Example** Instrument Run Log

17.5 Attachment 5 – Standard Certificate of Analysis

17.6 Attachment 6 – **DoD**-QSM LCS Control Limits

Attachment 1

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
dichlorodifluoromethane	1	85	87
chloromethane	1	50	52
vinyl chloride	1	62	64
bromomethane	1	94	96
chloroethane	1	64	66
trichlorofluoromethane	1	101	103
diethyl ether	1	74	45, 59
1,1-dichloroethene	1	96	61, 98
methylene chloride	1	84	49, 86
trans-1,2-dichloroethene	1	96	61, 98
1,1-dichloroethane	1	63	65, 83
2,2-dichloropropane	1	77	97
cis-1,2-dichloroethene	1	96	61, 98
bromochloromethane	1	128	49, 130
chloroform	1	83	85, 47
1,1,1-trichloroethane	1	97	99, 61
carbon tetrachloride	1	117	119, 121
1,1-dichloropropene	1	75	110, 77
benzene	1	78	77, 51
1,2-dichloroethane	1	62	98, 64
trichloroethene	1	130	95, 97
1,2-dichloropropane	1	63	39, 41
dibromomethane	1	174	93, 95
bromodichloromethane	1	83	85, 127
2-chloroethyl vinyl ether	1	63	65, 106
cis-1,3-dichloropropene	1	75	77, 110
acrolein	1	56	55, 38
iodomethane	1	142	127, 141
1,1,1-trichloro-2,2,2,-trifluoroethane	1	117	151, 153
1,1,2-trichloro-1,2,2,-trifluoroethane	1	85	101, 151
carbon disulfide	1	44	78, 76
acetone	1	43	58
3-chloropropene	1	39	41, 76
acetonitrile	1	41	39, 38
acrylonitrile	1	53	52, 51

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Attachment 1 (continued)

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
methyl-tert-butyl ether	1	73	41, 43
vinyl acetate	1	43	86
2-butanone	1	43	57, 72
propionitrile	1	54	55, 52
methacrylonitrile	1	41	39, 67
1,4-dioxane	1	88	58
methylmethacrylate	1	69	100, 41
Surrogate #1: dibromofluoromethane	1	113	111, 192
Surrogate #2: 1,2-dichloroethane-d_4	1	65	102, 67
4-methyl-2-pentanone	2	43	85, 100
toluene	2	92	91
trans-1,3-dichloropropene	2	75	77, 110
1,1,2-trichloroethane	2	97	83, 85
ethylmethacrylate	2	69	41, 99
tetrachloroethene	2	164	168, 129
1,3-dichloropropane	2	76	78
2-hexanone	2	43	58, 71
dibromochloromethane	2	129	127, 48
1,2-dibromoethane	2	107	109, 81
chlorobenzene	2	112	114, 77
1,1,1,2-tetrachloroethane	2	131	119, 133
ethylbenzene	2	106	91
m,p-xylene	2	106	91
o-xylene	2	106	91
styrene	2	104	78, 103
bromoform	2	173	175, 254
isopropyl benzene	2	105	120
bromobenzene	2	156	77,158
1,1,2,2-tetrachloroethane	2	83	85, 131
1,2,3-trichloropropane	2	110	75, 112
trans-1,4-dichloro-2-butene	2	53	88, 90
Surrogate #3: d_8-toluene	2	98	70, 100

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Attachment 1 (continued)

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
n-propyl benzene	3	91	120
2-chlorotoluene	3	126	91
4-chlorotoluene	3	126	91
1,2,4-trimethyl benzene	3	105	120
1,3,5-trimethyl benzene	3	105	120
Pentachloroethane	3	167	130, 165
sec-butyl benzene	3	105	134
1,2-dichlorobenzene	3	146	111, 148
1,3-dichlorobenzene	3	146	111, 148
1,4-dichlorobenzene	3	146	111, 148
n-butyl benzene	3	91	92, 134
tert-butyl benzene	3	119	91, 134
p-isopropyl toluene	3	119	134, 91
1,2-dibromo-3-chloropropane	3	157	75, 39
1,2,4-trichlorobenzene	3	180	182, 145
hexachlorobutadiene	3	225	223, 227
naphthalene	3	128	64, 51
1,2,3-trichlorobenzene	3	180	182, 145
Surrogate #4: 4-bromofluorobenzene	3	95	174, 176
Internal Standard #1: fluorobenzene	NA	96	70
Internal Standard #2: chlorobenzene-d_5	NA	117	82, 119
Internal Standard #3: 1,4-dichlorobenzene-d_4	NA	152	150

Attachment 2

8260B Reporting Limits

CAS#	Compound	5-mL (µg/L)	25-mL (µg/L)	5-grams (µg/Kg)
630-20-6	1,1,1,2-Tetrachloroethane	5	0.5	5
354-58-5	1,1,1-Trichloro-2,2,2-trifluoroethane	5	0.5	5
71-55-6	1,1,1,-Trichloroethane	5	0.5	5
79-34-5	1,1,2,2-Tetrachloroethane	5	0.5	5
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	5	0.5	5
79-00-5	1,1,2-Trichloroethane	5	0.5	5
75-34-3	1,1-Dichloroethane	5	0.5	5
75-35-4	1,1-Dichloroethene	5	0.5	5
563-58-6	1,1-Dichloropropene	5	0.5	5
87-61-6	1,2,3-Trichlorobenzene	5	0.5	5
96-18-4	1,2,3-Trichloropropane	5	0.5	5
120-82-1	1,2,4-Trichlorobenzene	5	0.5	5
95-63-6	1,2,4-Trimethylbenzene	5	0.5	5
96-12-8	1,2-Dibromo-3-Chloropropane	5	0.5	5
106-93-4	1,2-Dibromoethane	5	0.5	5
95-50-1	1,2-Dichlorobenzene	5	0.5	5
107-06-2	1,2-Dichloroethane	5	0.5	5
78-87-5	1,2-Dichloropropane	5	0.5	5
108-67-8	1,3,5-Trimethylbenzene	5	0.5	5
541-73-1	1,3-Dichlorobenzene	5	0.5	5
142-28-9	1,3-Dichloropropane	5	0.5	5
106-46-7	1,4-Dichlorobenzene	5	0.5	5
123-91-1	1,4-Dioxane	250	25	250
594-20-7	2,2-Dichloropropane	5	0.5	5
78-93-3	2-Butanone	12.5	2.5	12.5
110-75-8	2-Chloroethyl vinyl ether	5	5	5
95-49-8	2-Chlorotoluene	5	5	5
591-78-6	2-Hexanone	12.5	2.5	12.5
107-05-1	3-Chloropropene	5	5	5
106-43-4	4-Chlorotoluene	5	5	5
108-10-1	4-Methyl-2-pentanone	12.5	2.5	12.5
67-64-1	Acetone	12.5	2.5	12.5
75-05-8	Acetonitrile	5	5	5
107-02-8	Acrolein	50	5	50

N/A = Not Applicable to matrix

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Attachment 2 (continued)

8260B Reporting Limits

CAS#	Compound	5-mL µg/L	25-mL µg/L	5 grams µg/Kg
107-13-1	Acrylonitrile	50	5	50
71-43-2	Benzene	5	0.5	5
108-86-1	Bromobenzene	5	0.5	5
74-97-5	Bromochloromethane	5	0.5	5
75-27-4	Bromodichloromethane	5	0.5	5
75-25-2	Bromoform	5	0.5	5
74-83-9	Bromomethane	5	0.5	5
75-15-0	Carbon disulfide	5	0.5	5
56-23-5	Carbon Tetrachloride	5	0.5	5
108-90-7	Chlorobenzene	5	0.5	5
75-00-3	Chloroethane	5	0.5	5
67-66-3	Chloroform	5	0.5	5
74-87-3	Chloromethane	5	0.5	5
126-99-8	Chloroprene	5	0.5	5
156-59-2	cis-1,2-Dichloroethene	5	0.5	5
10061-01-5	cis-1,3-Dichloropropene	5	0.5	5
110-82-7	Cyclohexane	5	0.5	5
124-48-1	Dibromochloromethane	5	0.5	5
74-95-3	Dibromomethane	5	0.5	5
75-71-8	Dichlorodifluoromethane	5	0.5	5
100-41-4	Ethylbenzene	5	0.5	5
97-63-2	Ethylmethacrylate	50	5	50
87-68-3	Hexachlorobutadiene	5	0.5	5
74-88-4	Iodomethane	5	0.5	5
78-83-1	Isobutyl alcohol	250	25	250
89-82-8	Isopropyl Benzene	5	0.5	5
108-20-3	Isopropyl ether	5	0.5	5
108-38-3	m,p-Xylene	10	1	10
126-89-7	Methacrylonitrile	50	5	50
79-20-9	Methyl acetate	5	0.5	5
108-87-2	Methylcyclohexane	5	0.5	5
75-09-2	Methylene Chloride	5	0.5	5
80-62-6	Methylmethacrylate	50	5	50
1634-04-4	Methyl-tert-butyl ether	5	0.5	5
91-20-3	Naphthalene	5	0.5	5
104-51-8	n-Butyl Benzene	5	0.5	5
110-54-3	n-Hexane	N/A	0.5	N/A

N/A = Not Applicable to matrix

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Attachment 2 (continued)

8260B Reporting Limits

CAS#	Compound	5-mL µg/L	25-mL µg/L	5 grams µg/Kg
103-65-1	n-Propyl Benzene	5	0.5	5
95-47-6	o-Xylene	5	0.5	5
76-01-7	Pentachloroethane	5	0.5	5
99-87-6	p-Isopropyl Toluene	5	0.5	5
107-12-0	Propionitrile	250	0.5	250
135-98-8	sec-Butyl Benzene	5	0.5	5
100-42-5	Styrene	5	0.5	5
98-06-6	tert-Butyl Benzene	5	0.5	5
127-18-4	Tetrachloroethene	5	0.5	5
109-99-9	Tetrahydrofuran	N/A	2	20
108-88-3	Toluene	5	0.5	5
156-60-5	trans-1,2-Dichloroethene	5	0.5	5
10061-02-6	trans-1,3-Dichloropropene	5	0.5	5
110-57-6	trans-1,4-Dichloro-2-butene	100	20	100
79-01-6	Trichloroethene	5	0.5	5
75-69-4	Trichlorofluoromethane	5	0.5	5
108-05-4	Vinyl acetate	5	1	5
75-01-4	Vinyl Chloride	5	0.5	5
1330-20-7	Xylene (total)	15	1.5	15

N/A = Not Applicable to matrix

Attachment 3

Statistical Control Limits for the LCS

Compound	Percent Recovery Range	
	25 mL purge	**5 mL purge & med./high conc. Soil**
Dichlorodifluoromethane	50-150	50-150
Chloromethane	50-150	51-150
Vinyl chloride [2]	61-150	54-136
Bromomethane	50-150	59-150
Chloroethane	54-150	55-150
Trichlorofluoromethane	56-150	55-150
1,1-dichloroethene [1, 2]	74-143	70-130
Carbon disulfide	50-150	66-146
1,1,2-trichloro-1,2,2,-luoroethane	78-150	59-138
Methylene chloride [2]	50-139	72-123
trans-1,2-dichloroethene	50-137	39-130
Methyl-tert-butyl ether	68-134	62-135
1,1-dichloroethane [2]	59-138	70-126
cis-1,2-dichloroethene [2]	69-140	70-131
2-butanone [2]	65-134	64-127
Chloroform	67-147	65-133
1,1,1-trichloroethane	71-137	69-134
Carbon tetrachloride [2]	68-145	70-139
Benzene [1, 2]	68-138	69-130
1,2-dichloroethane	61-150	67-133
Trichloroethene [1, 2]	55-150	72-130
1,2-dichloropropane [2]	67-137	73-123
Bromodichloromethane [2]	73-142	71-126
cis-1,3-dichloropropene	74-134	63-136
4-methyl-2-pentanone	66-127	51-141
Toluene [1]	60-142	61-131
trans-1,3-dichloropropene	66-130	60-140
1,1,2-trichloroethane	68-130	63-131
Tetrachloroethene [2]	65-137	78-136
2-hexanone	53-140	50-143
Dibromochloromethane	68-137	70-129
1,2-dibromoethane	73-128	61-137
Chlorobenzene [1, 2]	68-129	76-121

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Attachment 3 (continued)

Statistical Control Limits for the LCS

Compound	**Percent Recovery Range**	
	25 mL purge	**5 mL purge & med./high conc. Soil**
Ethylbenzene [2]	67-127	68-131
Styrene [2]	66-139	73-127
Bromoform [2]	62-139	66-133
Isopropyl benzene	56-143	62-138
1,1,2,2-tetrachloroethane	63-122	63-135
1,3-dichlorobenzene	64-136	79-120
1,4-dichlorobenzene [2]	69-125	79-116
1,2-dichlorobenzene	71-127	62-143
1,2-dibromo-3-chloropropane	71-128	57-133
1,2,4-trichlorobenzene	52-147	57-142
Xylene (total)	60-140	61-150

Table displays statistical control limits calculated in 2002.

[1] Denotes Ohio VAP spike compounds. Each recovery must be within established control limits.

[2] SC DHEC spike compounds. Each of these analytes must be recovered within 70-130%.

Attachment 4

COMPUCHEM a Division of Liberty Analytical Corp DATE ____/____/____ INITIAL TIME OF TUNE_____ SHIFT/S(A)_____(B)_____(C)____
GC/MS VOLATILE RUN LOG TIME TUNE EXPIRES_____ LINKER /METHOD_____
COMPUCHEM LOGBOOK 11 NNN 29 (5972hp59) METHOD: 8260B SOM01.2 (Circle one) or Other: _____
MAINTENANCE_____

	REJECTED	FILE NAME	REPORTED	pH	Vial	DATE	TIME	Client ID	SDG#	INJ. VOL.*	DF+	CHEMIST	COMMENTS (ETC.)/ DISPOSITION
1						/ /							
2						/ /							
3						/ /							
4						/ /							
5						/ /							
6						/ /							
7						/ /							
8						/ /							
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22						/ /							
23						/ /							
24						/ /							

*On-column sample aliquot +Not applicable to soil matrix

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation

Tune (ID #7008) Lot No.: _____ Calibration Group Code / Lot No.: _____
Internal Standard Lot No.: _____ Surrogate Lot No.: _____

REVIEWED BY: _____ Date_____

4/05/10: jad

Note: This page subject to change without notification.

Attachment 5

TCL4 1&2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

Cat. No.: 30431 Lot No.: A027506
Description: 502.2 CAL 2000 Mega-Mix
Expiration Date[1]: August 2005 Storage: Freezer

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1,1-dichloroethane	75-34-3	99%	2,000 µg/ml	+/- .04%
1,1-dichloroethene	75-35-4	99%	2,000 µg/ml	+/- .04%
1,1-dichloropropene	563-58-6	99%	2,000 µg/ml	+/- .04%
1,1,1-trichloroethane	71-55-6	99%	2,000 µg/ml	+/- .04%
1,1,1,2-tetrachloroethane	630-20-6	99%	2,000 µg/ml	+/- .04%
1,1,2-trichloroethane	79-00-5	99%	2,000 µg/ml	+/- .04%
1,1,2,2-tetrachloroethane	79-34-5	99%	2,000 µg/ml	+/- .04%
1,2-dibromo-3-chloropropane	96-12-8	97%	2,000 µg/ml	+/- .04%
1,2-dibromoethane	106-93-4	99%	2,000 µg/ml	+/- .04%
1,2-dichlorobenzene	95-50-1	99%	2,000 µg/ml	+/- .04%
1,2-dichloroethane	107-06-2	99%	2,000 µg/ml	+/- .04%
1,2-dichloropropane	78-87-5	99%	2,000 µg/ml	+/- .04%
1,2,3-trichlorobenzene	87-61-6	99%	2,000 µg/ml	+/- .04%
1,2,3-trichloropropane	96-18-4	99%	2,000 µg/ml	+/- .04%
1,2,4-trichlorobenzene	120-82-1	99%	2,000 µg/ml	+/- .04%
1,2,4-trimethylbenzene	95-63-6	99%	2,000 µg/ml	+/- .04%
1,3-dichlorobenzene	541-73-1	99%	2,000 µg/ml	+/- .04%
1,3-dichloropropane	142-28-9	99%	2,000 µg/ml	+/- .04%
1,3,5-trimethylbenzene	108-67-8	99%	2,000 µg/ml	+/- .04%
1,4-dichlorobenzene	106-46-7	99%	2,000 µg/ml	+/- .04%
2-chlorotoluene	95-49-8	99%	2,000 µg/ml	+/- .04%
2,2-dichloropropane	594-20-7	99%	2,000 µg/ml	+/- .04%
4-chlorotoluene	106-43-4	99%	2,000 µg/ml	+/- .04%
p-isopropyltoluene	99-87-6	99%	2,000 µg/ml	+/- .04%
benzene	71-43-2	99%	2,000 µg/ml	+/- .04%
bromobenzene	108-86-1	99%	2,000 µg/ml	+/- .04%
bromochloromethane	74-97-5	99%	2,000 µg/ml	+/- .04%

[1]Expiration date of the unopened ampul stored at recommended temperature.

[2]Listed in alphabetical order.

[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.

[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).

[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

Attachment 5 (continued)

TCL4 1&2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS (cont.)

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

PAGE 2

Cat. No.: 30431 Lot No.: A027506

Description: 502.2 CAL 2000 Mega-Mix

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
bromodichloromethane	75-27-4	99%	2,000 µg/ml	+/- .04%
bromoform	75-25-2	99%	2,000 µg/ml	+/- .04%
carbon tetrachloride	56-23-5	99%	2,000 µg/ml	+/- .04%
chlorobenzene	108-90-7	99%	2,000 µg/ml	+/- .04%
chloroform	67-66-3	99%	2,000 µg/ml	+/- .04%
cis-1,2-dichloroethene	156-59-2	99%	2,000 µg/ml	+/- .04%
cis-1,3-dichloropropene	10061-01-5	99%	2,000 µg/ml	+/- .04%
dibromochloromethane	124-48-1	99%	2,000 µg/ml	+/- .04%
dibromomethane	74-95-3	99%	2,000 µg/ml	+/- .04%
ethylbenzene	100-41-4	99%	2,000 µg/ml	+/- .04%
hexachlorobutadiene	87-68-3	99%	2,000 µg/ml	+/- .04%
isopropylbenzene	98-82-8	99%	2,000 µg/ml	+/- .04%
methylene chloride	75-09-2	99%	2,000 µg/ml	+/- .04%
m-xylene	108-38-3	99%	2,000 µg/ml	+/- .04%
naphthalene	91-20-3	99%	2,000 µg/ml	+/- .04%
n-butylbenzene	104-51-8	99%	2,000 µg/ml	+/- .04%
n-propylbenzene	103-65-1	99%	2,000 µg/ml	+/- .04%
o-xylene	95-47-6	99%	2,000 µg/ml	+/- .04%
p-xylene	106-42-3	99%	2,000 µg/ml	+/- .04%
sec-butylbenzene	135-98-8	99%	2,000 µg/ml	+/- .04%
styrene	100-42-5	99%	2,000 µg/ml	+/- .04%
tetrachloroethene	127-18-4	99%	2,000 µg/ml	+/- .04%
tert-butylbenzene	98-06-6	99%	2,000 µg/ml	+/- .04%
toluene	108-88-3	99%	2,000 µg/ml	+/- .04%
trans-1,2-dichloroethene	156-60-5	99%	2,000 µg/ml	+/- .04%
trans-1,3-dichloropropene	10061-02-6	97%	2,000 µg/ml	+/- .04%
trichloroethene	79-01-6	99%	2,000 µg/ml	+/- .04%
Solvent: purge & trap methanol	67-56-1	99%		

[1]*Expiration date of the unopened ampul stored at recommended temperature.*

[2]*Listed in alphabetical order.*

[3]*Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.*

[4]*Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).*

Attachment 5 (continued)

TCL4 - GASES HIGH

RESTEK

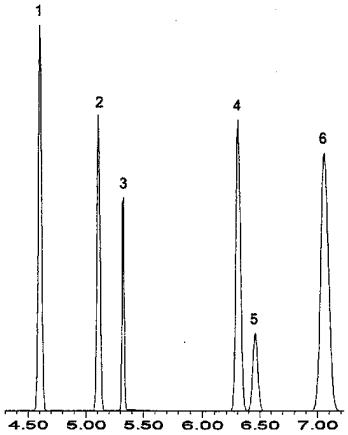
CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY--READ MSDS PRIOR TO USE.

Cat. No.: 30042 Lot No.: A028050
Description: 502.2 Calibration Mix #1
Expiration Date[1]: March 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	Dichlorodifluoromethane	75-71-8	99%	1,997 µg/ml	± 1.9%
2	Chloromethane	74-87-3	99%	2,003 µg/ml	± 1.3%
3	Vinyl chloride	75-01-4	99%	2,003 µg/ml	± 1.4%
4	Bromomethane	74-83-9	99%	1,997 µg/ml	± 1.3%
5	Chloroethane	75-00-3	99%	2,003 µg/ml	± 1.5%
6	Trichlorofluoromethane	75-69-4	99%	2,000 µg/ml	± 0.3%
Solvent:	Purge and trap Methanol	67-56-1	99%		



Column: 105m .32mm 1.8µm
Rtx-502.2 (cat.#10921)
Carrier gas: helium @ 2.2 ml/min.
Temp. program: 50°C (hold 12 min.)
Isothermal
Inj. temp.: 240°C
Det. temp.: 240°C
Detector type: MSD

John Lidgett
John Lidgett—QA Analyst

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

[1] *Expiration date of the unopened ampul stored at recommended temperature.*
[2] *Listed in alphabetical order.*
[3] *Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.*
[4] *Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).*
[5] *Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.*

Attachment 5 (continued)

TCL4- KETONES HIGH

AccuStandard Inc.

125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: S-9863-R1
DESCRIPTION: Custom VOA Std #2
LOT #: B3110072
SOLVENT: Methanol

EXPIRATION: Feb 9, 2004

See reverse for additional certification information.

This product is guaranteed accurate to + 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetone	67-64-1	99.9	1261 ± 50.44	1260
Methyl ethyl ketone	78-93-3	100	1261 ± 50.44	1261
4-methyl-2-pentanone	108-10-1	99.4	1261 ± 50.44	1253
2-hexanone	591-78-6	100	1261 ± 50.44	1261
Vinyl acetate	108-05-4	99.9	500.8 ± 20.03	500.3
2-Chloroethylvinyl ether	110-75-8	99.7	500.3 ± 20.01	498.8
Acrolein	107-02-8	90.0	5575 * ± 223.00	5018
Acrylonitrile	107-13-1	99.9	5000 ± 200.00	4995

8 Components

1. All weights are traceable through NIST, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty $u_c(y)$. It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is $U_c(y)$ *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-00
Rev. 11/0

Attachment 5 (continued)

TCL4-APPIX H1

AccuStandard Inc.

125 Market Street
New Haven, CT 06513
USA

Ph: 203-786-5290
Fax: 203-786-5287
E-mail: usa@accustandard.com
www.accustandard.com

CERTIFICATE OF ANALYSIS

PRODUCT: S-9862-R1
EXPIRATION: Feb 27, 2004
DESCRIPTION: Custom VOA Std #1
LOT #: B3110080 **See reverse for additional certification information.**
SOLVENT: Methanol

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetonitrile	75-05-8	99.9	500.3 ±20.01	499.8
Allyl chloride	107-05-1	99.6	500.0 ±20.00	498.0
1-Chlorohexane	544-10-5	99.0	500.3 ±20.01	495.3
Chloroprene	126-99-8	98	500.0 ±20.00	490.0
trans-1,4-Dichloro-2-butene	110-57-6	97.4	25,670 * ±1026.80	25003
p-Dioxane	123-91-1	99.2	25,010 ±1000.40	24810
Ethyl methacrylate	97-63-2	99.0	5010 ±200.40	4960
Isobutyl alcohol	78-83-1	100	25,010 ±1000.40	25010
Methacrylonitrile	126-98-7	99.9	5010 ±200.40	5005
Methyl iodide	74-88-4	100	500.3 ±20.01	500.3
Methyl methacrylate	80-62-6	99.8	5003 ±200.12	4993
Pentachloroethane	76-01-7	97.3	514.0 * ±20.56	500.1
Propionitrile	107-12-0	99.9	25,010 ±1000.40	24985

13 Components

1. All weights are traceable through NIST, Test No. 822/254480.
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is the Combined Uncertainty $u_c(y)$. It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is $U_c(y)$ *K where K is the coverage factor at the 95% confidence level (K=2). Values reported above are Expanded Combined Uncertainty.
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

This product was manufactured to meet the quality system requirements of ISO 9001

QR-ORG/INO-00

Attachment 5 (continued)

RESTEK

CERTIFICATE OF ANALYSIS

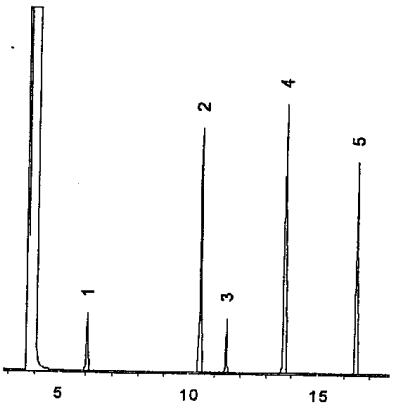
110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY—READ MSDS PRIOR TO USE.

Cat. No.: 30005 Lot No.: A019743
Description: VOA Matrix Spike Mix
Expiration Date[1]: January 2005

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	1,1-dichloroethene	75-35-4	99%	2,500 µg/ml	± 0.2%
2	benzene	71-43-2	99%	2,500 µg/ml	± 0.2%
3	trichloroethene	79-01-6	99%	2,500 µg/ml	± 0.2%
4	toluene	108-88-3	99%	2,500 µg/ml	± 0.2%
5	chlorobenzene	108-90-7	99%	2,500 µg/ml	± 0.2%
Solvent:	purge and trap methanol	67-56-1	99%		

Column: 105m .53mm 3.0µm
Rtx®-502.2 (cat.#10910)
Carrier gas: hydrogen @ 40cm/sec
Temp. program: 40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)
Inj. temp.: 200°C
Det. temp.: 250°C
Detector type: FID



John Lidgett
John Lidgett—QA Analyst

[1]Expiration date of the unopened ampul stored at recommended temperature.
[2]Listed in alphabetical order.
[3]As determined by capillary GC/FID unless otherwise noted. Value rounded to the nearest LOWER whole percentage. In addition to GC/FID, chemical identity and purity are confirmed using 2 or more of the following: GC/MS, solid probe MS, GC/ECD, GC/FPD, GC/NPD, GC/TC, HPLC, DSC, FTIR, melting point, refractive index, and Karl Fisher.
[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

Attachment 6

Solid and Aqueous LCS Control Limits for 8260B Required by the DOD-QSM

CAS#	Compound	Water CL	Water ME	Soil CL	Soil ME
630-20-6	1,1,1,2-Tetrachloroethane	80-130	75-135	75-125	65-135
354-58-5	1,1,1-Trichloro-2,2,2-trifluoroethane	N/A	N/A	N/A	N/A
71-55-6	1,1,1,-Trichloroethane	65-130	55-140	70-135	55-145
79-34-5	1,1,2,2-Tetrachloroethane	75-125	65-135	55-130	40-145
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	N/A	N/A	N/A	N/A
79-00-5	1,1,2-Trichloroethane	75-125	65-135	60-125	50-140
75-34-3	1,1-Dichloroethane	70-135	60-145	75-125	65-135
75-35-4	1,1-Dichloroethene	70-130	55-140	65-135	55-150
563-58-6	1,1-Dichloropropene	75-130	65-140	70-135	60-145
87-61-6	1,2,3-Trichlorobenzene	55-140	45-155	60-135	50-145
96-18-4	1,2,3-Trichloropropane	75-125	65-130	65-130	50-140
120-82-1	1,2,4-Trichlorobenzene	65-135	55-145	65-130	55-140
95-63-6	1,2,4-Trimethylbenzene	75-130	65-140	65-135	55-145
96-12-8	1,2-Dibromo-3-Chloropropane	50-130	35-145	40-135	25-150
106-93-4	1,2-Dibromoethane	80-120	75-125	70-125	60-135
95-50-1	1,2-Dichlorobenzene	70-120	60-130	75-120	65-125
107-06-2	1,2-Dichloroethane	70-130	60-140	70-135	60-145
78-87-5	1,2-Dichloropropane	75-125	65-135	70-120	65-125
108-67-8	1,3,5-Trimethylbenzene	75-130	65-140	65-135	55-145
541-73-1	1,3-Dichlorobenzene	75-125	65-130	70-125	65-135
142-28-9	1,3-Dichloropropane	75-125	65-135	75-125	70-130
106-46-7	1,4-Dichlorobenzene	75-125	65-130	70-125	65-135
123-91-1	1,4-Dioxane	N/A	N/A	N/A	N/A
594-20-7	2,2-Dichloropropane	70-135	60-150	65-135	55-145
78-93-3	2-Butanone	30-150	10-170	30-160	10-180
110-75-8	2-Chloroethyl vinyl ether	N/A	N/A	N/A	N/A
95-49-8	2-Chlorotoluene	75-125	65-135	70-130	60-140
591-78-6	2-Hexanone	55-130	45-140	45-145	30-160
107-05-1	3-Chloropropene	N/A	N/A	N/A	N/A
106-43-4	4-Chlorotoluene	75-130	65-135	75-125	65-135
108-10-1	4-Methyl-2-pentanone	60-135	45-145	45-130	30-165
67-64-1	Acetone	40-140	20-160	20-160	10-180
75-05-8	Acetonitrile	N/A	N/A	N/A	N/A
107-02-8	Acrolein	N/A	N/A	N/A	N/A

CL =control limit
ME = marginal exceedances
N/A = not included in DOD list

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Attachment 6

Solid and Aqueous LCS Control Limits for 8260B Required by the **DoD**-QSM

CAS#	Compound	Water CL	Water ME	Soil CL	Soil ME
107-13-1	Acrylonitrile	N/A	N/A	N/A	N/A
71-43-2	Benzene	80-120	75-130	75-125	65-135
108-86-1	Bromobenzene	75-125	70-130	65-120	55-130
74-97-5	Bromochloromethane	65-130	55-140	70-125	60-135
75-27-4	Bromodichloromethane	75-120	70-130	70-130	60-135
75-25-2	Bromoform	70-130	60-140	55-135	45-150
74-83-9	Bromomethane	30-145	10-165	30-160	10-180
75-15-0	Carbon disulfide	35-160	15-185	45-160	30-180
56-23-5	Carbon Tetrachloride	65-140	55-150	65-135	55-145
108-90-7	Chlorobenzene	80-120	75-130	75-125	65-130
75-00-3	Chloroethane	60-135	50-145	40-155	20-175
67-66-3	Chloroform	65-135	50-150	70-125	65-135
74-87-3	Chloromethane	40-125	25-140	50-130	40-140
126-99-8	Chloroprene	N/A	N/A	N/A	N/A
156-59-2	cis-1,2-Dichloroethene	70-125	60-135	65-125	55-135
10061-01-5	cis-1,3-Dichloropropene	70-130	60-140	70-125	65-135
110-82-7	Cyclohexane	N/A	N/A	N/A	N/A
124-48-1	Dibromochloromethane	60-135	45-145	65-130	55-140
74-95-3	Dibromomethane	75-125	65-135	75-130	65-135
75-71-8	Dichlorodifluoromethane	30-155	10-175	35-135	15-155
100-41-4	Ethylbenzene	75-125	65-135	75-125	65-135
97-63-2	Ethylmethacrylate	N/A	N/A	N/A	N/A
87-68-3	Hexachlorobutadiene	50-140	35-160	55-140	40-155
74-88-4	Iodomethane	N/A	N/A	N/A	N/A
78-83-1	Isobutyl alcohol	N/A	N/A	N/A	N/A
89-82-8	Isopropylbenzene	75-125	65-135	75-130	70-140
108-20-3	Isopropyl ether	N/A	N/A	N/A	N/A
108-38-3	m,p-Xylene	75-130	65-135	80-125	70-135
126-89-7	Methacrylonitrile	N/A	N/A	N/A	N/A
79-20-9	Methyl acetate	N/A	N/A	N/A	N/A
108-87-2	Methylcyclohexane	N/A	N/A	N/A	N/A
75-09-2	Methylene Chloride	55-140	40-155	55-140	40-155
80-62-6	Methylmethacrylate	N/A	N/A	N/A	N/A
1634-04-4	Methyl-tert-butyl ether	65-125	55-135	N/A	N/A
91-20-3	Naphthalene	55-140	40-150	40-125	25-140
104-51-8	n-Butylbenzene	70-135	55-150	65-140	50-150

CL =control limit
ME = marginal exceedances
N/A = not included in DOD list

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If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 6

Solid and Aqueous LCS Control Limits for 8260B Required by the **DoD**-QSM

CAS#	Compound	Water CL	Water ME	Soil CL	Soil ME
110-54-3	n-Hexane	N/A	N/A	N/A	N/A
103-65-1	n-Propylbenzene	70-130	65-140	65-135	50-145
95-47-6	o-Xylene	80-120	75-130	75-125	70-135
76-01-7	Pentachloroethane	N/A	N/A	N/A	N/A
99-87-6	p-Isopropyltoluene	75-130	64-140	75-135	65-140
107-12-0	Propionitrile	N/A	N/A	N/A	N/A
135-98-8	sec-Butylbenzene	70-125	65-135	65-130	50-145
100-42-5	Styrene	65-135	55-145	75-125	65-135
98-06-6	tert-Butylbenzene	70-130	60-140	65-130	55-145
127-18-4	Tetrachloroethene	45-150	25-165	65-140	55-150
109-99-9	Tetrahydrofuran	N/A	N/A	N/A	N/A
108-88-3	Toluene	75-120	70-130	70-125	60-135
156-60-5	trans-1,2-Dichloroethene	60-140	45-150	65-135	55-145
10061-02-6	trans-1,3-Dichloropropene	55-140	40-155	65-125	55-140
110-57-6	trans-1,4-Dichloro-2-butene	N/A	N/A	N/A	N/A
79-01-6	Trichloroethene	70-125	60-135	75-125	70-130
75-69-4	Trichlorofluoromethane	60-145	45-160	25-185	10-215
108-05-4	Vinyl acetate	N/A	N/A	N/A	N/A
75-01-4	Vinyl Chloride	50-145	35-165	60-125	45-140
1330-20-7	Xylene (total)	N/A	N/A	N/A	N/A

CL =control limit
ME = marginal exceedances
N/A = not included on DOD list

SOP 1.3.2.4

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP481B SOP Section #: 1.3.2.4 Revision #: 13

SOP Title: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 Method 8260B

Effective date: (QA fills in) 4/8/11

- Procedure prepared by: [signature] Date: 4-7-11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 4-8-11
- Reason for change: Addition of DoD parameters and protocols, addition of LIMS information
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, 12/96, Method 8260B, Method 5035A; DoD-QSM, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: [signature] NA [signature] 4/8/11 Date: 4-3-11 NA [signature] 4/8/11

Annual Review—Signature: [signature] Date: 6-30-11

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 481B: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 Method 8260B

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Instrument Procedure 481B: GC/MS Analysis of Low Concentration Volatiles in Soil/Sediment/Sludge Samples by SW-846 Method 8260B

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the procedure for the purge and trap GC/MS analysis of solid (soil, sediment and sludge) samples following Method 8260B and incorporating Method 5035A. The method is applicable to a wide range of organic compounds. Target compounds that may be analyzed by this method are listed in Attachment 1, along with their associated internal standards and quantitation ions. Note, however, that many of these compounds are not routinely analyzed.

The reporting limit is the low-level calibration standard concentration. Reporting limits for this method are shown in Attachment 3.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Low concentration level volatiles samples are analyzed using a closed system purge and trap technique (Method 5035A). Field samples may be collected in vials containing a preservative solution (sodium bisulfate), immediately sealed and shipped to the laboratory at 4 °C ± 2 °C. Normally, an EnCore sampling device (or equivalent) is used to obtain a 5g sample that does not contain headspace. When the EnCore device is used, it is sent refrigerated to the laboratory and, within 48 hours of sampling, the contents of the Encore device must be transferred to a closed-system purge and trap **vial** and placed in a freezer at – 10 to – 20 °C.

A special autosampler is used for the analysis. This device allows the sample vial to remain closed while reagent water and a solution containing internal standards and surrogates are injected by a double-walled needle through the septum. The needle used to pierce the septum to deliver the water, internal standards, and surrogates is then the source of an inert gas which allows adequate mixing through the bottom of the needle. The same needle has entrance holes located above the sample/water level to collect and

transfer the headspace onto a sorbent column, where the purgeables are trapped. The autosampler also provides for heating the sample vial to 40 °C while the contents are being mixed and the sample constituents purged and trapped. After purging is completed, the sorbent column is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatograph (GC) wide-bore capillary column. The GC is temperature-programmed to separate the purgeables that are then detected with a mass spectrometer (MS).

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic

methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – µg/kg

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 **DoD**-QSM – Department of Defense Quality Systems Manual

3.10 Marginal Exceedance – Beyond the LCS control limit but within the marginal exceedance limits (set at 4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing.

3.11 Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.12 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Impurities in the purge gas or methanol, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. Gas lines from the gas tanks to the instrument must be either stainless steel or copper tubing. Non-polytetrafluoroethylene (PTFE) thread sealants, or flow controllers with rubber components are not to be used. When potential interfering peaks are noted in laboratory method blanks, one or more of the following corrective actions may be necessary to reduce solvent contamination: purging the methanol used to prepare standard solutions, purging the DI water with helium or nitrogen, changing the purge gas source, and regenerating the molecular sieve purge gas filter.

4.2 Samples can be contaminated by diffusion of purgeable organics (particularly methylene chloride, fluorocarbons, and other common laboratory solvents) through the septum seal into the sample during storage and handling. Therefore, these samples are stored in the GC/MS VOA laboratory freezer; separate from laboratory standards, and they must be analyzed in a room in which the atmosphere is demonstrated to be free of all potential contaminants that will interfere with the analysis. Because methylene chloride will permeate PTFE tubing, all GC carrier gas lines and purge gas plumbing are to be constructed from stainless steel or copper tubing.

4.3 Contamination by carryover can occur whenever a sample is analyzed after a sample that contains high levels of organic compounds. Whenever an unusually concentrated sample is encountered, it must either be followed by analysis of an instrument blank or the next sample must be closely monitored to check for cross-contamination. For samples containing large amounts of water soluble materials, high boiling compounds, or high purgeable levels, it may be necessary to clean the purge and trap apparatus by purging a 10-20% methanol solution, followed by baking the purge and trap apparatus and the analysis of an instrument blank to confirm that the system is free from contamination. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

5.0 Safety

5.1 The toxicity and carcinogenicity of many chemicals used in this method have not been precisely determined: each chemical should be treated as a potential health hazard. Exposure to these chemicals should be minimized. Preparation of calibration standards, blanks, and samples is performed in a fume hood to minimize risk.

5.2 The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,2- dichloroethane, 1,1,2,2-tetrachloroethane, 1,1,2- trichloroethane, chloroform, 1,2-dibromoethane, trichloroethene, and vinyl chloride.

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff members are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 5, 10, and 25 mL Gastight Syringes with Luerlock tips

6.2 1 and 10 µL Hamilton syringes

6.3 Assorted volumetric flasks ranging from 50 mL to 1000 mL

6.4 1 mL mininert vials

6.5 40 mL screw-top, PTFE-lined, septum-sealed vials

6.6 Magnetic stirring bars

6.7 Analytical Column

6.7.1 Supelco SPB-624 60-m, 0.32mm ID with 1.8 um film thickness

6.7.2 Descriptions of alternative capillary columns are presented in EPA Contract Laboratory Program (CLP) Statement of Work (SOW)

documents. Columns other than those listed may be used if equivalency is demonstrated. Specifications for equivalency are also presented in the SOW documents. The equivalency documentation must be maintained and made available during on-site audits

6.8 Purge and Trap Autosampler System

6.8.1 Tekmar LSC

6.8.1.1 Tekmar, LSC 3000, LCS 3100 and Velocity XPT with glass frit bottom liquid sample purging vessel and Luerlock valve

6.8.1.2 The absorption trap must be at least 25 cm long and have an internal diameter of at least 0.105 inches (0.2667 cm).

6.8.1.3 The Supelco K (VOCARB 3000) trap consists of the following:

- Carbopack B, 10 cm
- Carboxen-1000, 6 cm
- Carboxen-1001, 1 cm

6.8.1.4 Alternate sorbent traps may be used if they meet the QC acceptance criteria. Performance documentation must be maintained and made available during an on-site audit.

6.8.2 Archon ALS

6.8.2.1 The Archon Model 5100, 4552 Purge and trap autosampler, interfaces directly to a Tekmar Purge and Trap Concentrators.

6.8.2.2 The autosampler is designed for soil samples and utilizes 40 mL VOA vials with low bleed Teflon septa. All samples, water and soil, are purged in situ in the VOA vials.

6.8.2.3 The Archon ALS has the capacity of up to 51 vials.

6.9 GCMS

6.9.1 Hewlett Packard 5890 Series

6.9.2 Hewlett Packard 6890 Series

6.9.3 Agilent 6890N Series

6.9.4 Hewlett Packard 5972 Mass Selective Detector (MSD)

6.9.5 Hewlett Packard 5973 MSD

6.9.6 Hewlett Packard 6890MSD

6.9.7 Agilent Technologies 5975 MSD

6.9.8 Interface (GC to mass spectrometer)

6.9.8.1 Direct capillary interface at 225 °C is used for Hewlett Packard MSDs.

6.10 Data system

6.10.1 The Hewlett Packard GC/MS systems utilize ChemServer software, for data acquisition.

6.10.2 For data processing, CompuChem uses the Hewlett Packard HP 9000 series 735 Unix Workstation employing Target3 and Envision software by Thru-Put Systems.

6.10.3 The reference library used is the NIST Library (NIST2005), purchased from ThermoFisher Scientific, plus updates and revisions.

6.11 Data storage

Data are stored on the Target3 Unix Workstation. The Workstation is backed up incrementally on a daily basis.

6.12 Element DataSystem® LIMS computer software by Promium is used for sample management and report generation.

7.0 <u>Reagents and Standards</u>

Note: Detailed standard preparation information may be found in the Standard Preparation Logbook 11D(4), "GCMS VOA Standard Preparation Logbook", located in the Volatile Laboratory or in LIMS.

All standards are prepared in the Volatile Organic Laboratory. Stock Standards are stored separately from samples in Teflon®- sealed screw-cap bottles with zero headspace at -10 to -20 ^{o}C in the freezer units in the instrument laboratory when not in use. Protect the standards from light. Standards for gases usually need to be replaced after one week or as recommended by the manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases must be monitored and fresh standards prepared if a 20% drift is experienced. These standards need to be replaced after six months or as recommended by the manufacturer, unless the acceptability of the standard can be documented. 2-chloroethyl vinyl ether (CEVE) and styrene may have to be prepared more frequently. Secondary dilutions of Stock Standards must be checked frequently for signs of degradation or evaporation, especially just prior to their use in preparing the working calibration standards. Working standards must be prepared just prior to analysis unless they are to be purged by an autosampler. When an autosampler is used, the standards may be kept up to 12 hours in purge vessels connected via the autosampler to the purge and trap device.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: **All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.** All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent Water-All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is subsequently purged with an inert gas and demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methanol (B&J Scientific, purge and trap grade)

7.3 Sodium bisulfate, $NaHSO_4$ - ACS reagent grade or equivalent

7.4 Tuning Standard

7.4.1 Bromofluorobenzene - Standard ID# 7008 at 25 µg/mL. Two µL, yielding 50 **η**g on column, are injected onto the column every 12 hours.

7.4.1.1 Prepare the standard by adding 50 µL Restek VOA Tuning Mix 5000 µg/mL) to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.4.1.2 Prepare this standard monthly.

7.5 Calibration Standards

7.5.1 For the initial calibration, the internal standard solution is added automatically by the Archon Purge and Trap Autosampler. For all subsequent analyses, both the internal standard and the surrogate solutions are added automatically by the Archon autosampler.

Standard Preparation

Std. ID TCL4-High	**005 µg/kg**	**010 µg/kg**	**020 µg/kg**	**050* µg/kg**	**100 µg/kg**	**200 µg/kg**
8260 S.S.	10.0 µL	20.0 µL	50.0 µL	100.0 µL	200.0 µL	400.0 µL
TCL4-1&2	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL
TCL4-gases	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL
TCL4-ketones	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL
TCL4-AppIX	1.0 µL	2.0 µL	4.0 µL	10.0 µL	20.0 µL	40.0 µL

*Continuing Calibration level

7.5.1.1 To prepare the standards at the concentrations shown in the column headers of the preceding table above, add the µL amount of standard shown to a 100 mL volumetric flask containing DI water, then bring up to volume.

Alternatively, the standards may be prepared at the above concentrations by diluting the 200 µg/kg standard directly into the purge and trap impingers. Dilute as follows:

- For a 100 µg/kg standard, add 2.5 mL to 5 mL DI water;
- For a 50 µg/kg standard, add 1.25 mL to 5 mL DI water;
- For a 20 µg/kg standard, add 0.50 mL to 5 mL DI water;

- For a 10 µg/kg standard, add 0.25 mL to 5 mL DI water;
- For a 5 µg/kg standard, add 0.125 mL to 5 ml DI water.

7.5.1.2 The concentrations of the compounds in the TCL4-1&2 High standard are 500 µg/mL. See Attachment 5 for the composition of this standard.

7.5.1.2.1 Prepare the standard by adding 1.25mL Restek 502.2 VOA 2000 MegaMix (2000 µg/mL) and 1.25 mL of Restek Custom 502.2 Additions Mix (2000 µg/mL) to an amount of purge and trap grade methanol in a 5 mL volumetric flask and bring to volume.

7.5.1.2.2 Prepare this standard every three months.

7.5.1.3 The concentrations of the compounds in the TCL4-gases High standard are 500 µg/mL. See Attachment 5 for the composition of this standard.

7.5.1.3.1 Prepare each standard by adding 1.25mL Restek 502.2 Calibration Mix #1 (2000 µg/mL) to an amount of purge and trap grade methanol in a 5 mL volumetric flask and bring to volume.

7.5.1.3.2 The method states that this standard usually needs to be replaced weekly, unless the standard manufacturer recommends otherwise, or unless the acceptability of the standard can be documented. This standard has generally proven to be more stable in the laboratory. The gas standard can be used for longer than a week if the gases in the continuing calibration (CCV) standard meet the CCV requirements when compared to the initial calibration standards that contain a gas standard that has been prepared within a one-week holding time. Prepare this standard monthly, or more frequently as need dictates, or when degradation is evident rendering the standard unacceptable.

7.5.1.4 The concentrations of the compounds in the TCL4-ketones standard are 500 µg/mL, 1250 µg/mL, or 5000 µg/mL. See Attachment 5 for the composition of this standard.

7.5.1.4.1 Prepare the standard by transferring AccuStandard custom VOA Mix #2 to a mininert vial.

7.5.1.4.2 Replenish this standard as needed and replace every three months.

7.5.1.5 The concentrations of the compounds in the TCL4-AppIX standard are 500 µg/mL, 5000 µg/mL, or 25000 µg/mL. See Attachment 5 for the composition of this standard.

7.5.1.5.1 Prepare the standard by transferring AccuStandard custom VOA Mix #1 to a mininert vial.

7.5.1.5.2 Replenish this standard as needed and replace every three months.

7.6 Initial Calibration Verification Standard (also the Full Spike LCS, MS, and MSD Stock Standard)

7.6.1 The ICV contains the full list of target analytes at the same concentration as the continuing calibration verification (CCV) standard. See Attachment 7.

7.6.2 The initial calibration curve must be verified using a standard from a second source. The laboratory purchases the initial calibration verification (ICV) standards from a different vendor than the ones used for the calibration standards. This is prepared in the same manner as described above. See Section 7.5 or Standard Preparation Logbook 11D(4). Also See Attachment 7.

7.6.2.1 The concentrations of the compounds in Second source TCL4-1 & 2 High standard are 500 µg/mL.

7.6.2.1.1 Prepare the standard by adding 1.25 mL of AccuStandard M-502A-R-10X and 1.25 mL of AccuStandard S-13035 to an amount of purge and trap grade methanol in a 5 mL volumetric flask and bring to volume with purge and trap grade methanol.

7.6.2.2 Prepare this standard every three months or when degradation is evident rendering the standard unacceptable.

7.6.2.3 The concentrations of the compounds in the second source TCL4-gases standard are 500 µg/mL. See Attachment 7.

7.6.2.3.1 Prepare each standard by adding 1.25 mL of AccuStandard M-502B-10X to an amount of purge and trap grade methanol in a 5 mL volumetric flask and bring to volume.

7.6.2.3.2 The method states that this standard usually needs to be replaced weekly, unless the standard manufacturer recommends otherwise, or unless the acceptability of the standard can be documented. This standard has generally proven to be more stable in the laboratory. The gas standard can be used for longer than a week if the gases in the continuing calibration (CCV) standard meet the CCV requirements when compared to the initial calibration standards that contain a gas standard that has been prepared within a one week holding time. Prepare this standard monthly, or more frequently as need dictates, or when degradation is evident rendering the standard unacceptable.

7.6.3 The concentrations of the compounds in the second source TCL4-ketones standard are 500 µg/mL, 1250 µg/mL, or 5000 µg/mL. See Attachment 7.

7.6.3.1 Prepare each standard by adding 800µL of NSI custom Acrolein standard Q-6087-0, 800µL of Restek Custom Vinyl Acetate standard 560347, and 800µL of Restek Custom Ketone Plus to clean dry 2.5 mL-miniert and invert to mix.

7.6.3.2 Prepare this standard every three months or when degradation is evident rendering the standard unacceptable.

7.6.4 The concentrations of the compounds in the second source TCL4-AppIX standard are 500 µg/mL, 5000 µg/mL, or 25000 µg/mL. See Attachment 7.

7.6.4.1 Prepare the standard by transferring Restek custom VOA Standard #1 to a mininert vial.

7.6.4.2 Replenish this standard as needed and replace every three months or when degradation is evident rendering the standard unacceptable.

7.7 Internal Standards

Compounds in 8260 I.S. (Internal standard) at a concentration of 50 µg/mL (250 µg/mL for the Archon)

- fluorobenzene
- D5-chlorobenzene
- D4-1,4-dichlorobenzene

7.7.1 Prepare the internal standard spiking solution by adding 0.2 mL Restek 8260A ampulated internal standard mix (2500 µg/mL) to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.7.2 Prepare this standard every three months.

7.8 Surrogate Standard

Compounds in 8260 S.S. (Surrogate Standard) at a concentration of 50 µg/mL (250 µg/mL for the Archon)

- dibromofluoromethane
- D4-1,2-dichloroethane
- D8-toluene
- 4-bromofluorobenzene

7.8.1 Prepare the surrogate spiking solution by adding 0.2 mL Restek 8260A ampulated surrogate mix (2500 µg/mL) to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.8.2 Prepare this standard every three months.

7.9 Short List Spike Standard

Compounds in 8260 Spiking Mixture (1001C) at a concentration of 25 µg/mL:

- 1,1-dichloroethene
- trichloroethene

- benzene
- toluene
- chlorobenzene

Note: The spiking cocktail is project dependent. This spiking mixture can also be used as a Laboratory Control Sample (LCS) spike. Some projects may require full analyte spike, and in that case, the standard used for the full analyte spike LCS is the ICV. For some programs, the CCV may be used in the place of the LCS.

7.9.1 Prepare this standard by adding 100 µ**L** Restek VOA Matrix Spike Mix to an amount of purge and trap grade methanol in a 10 mL volumetric flask and bring to volume.

7.9.2 Prepare this standard every six months.

8.0 Sample Preservation and Storage

8.1 Samples are preserved, and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All samples must be analyzed within 14 days of collection.

8.3 Prior to analysis, all samples must be stored in the freezer unit in the laboratory at a temperature of -10 to -20 °C. After analysis, samples are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 Surrogate compounds are added to all field and QC samples prior to analysis. Surrogates are used to assess the efficiency of the analytical system.

9.1.2 Surrogate compounds must meet recovery criteria as shown below:

Surrogate Compound	**Soil % Recovery Range**
Dibromofluoromethane	71-141
D4-1,2-dichloroethane	70-139
D8-toluene	72-123

4-bromofluorobenzene	63-131

9.1.2.1 The same surrogates and recovery criteria are to be used for samples submitted to meet the regulatory requirements of the State of South Carolina.

9.1.2.2 The following table contains the surrogate recovery limits required by the DOD-QSM.

Surrogate Compound	**Soil % Recovery Range**
Dibromofluoromethane	N/A
D4-1,2-dichloroethane	N/A
D8-toluene	85-115
4-bromofluorobenzene	85-120

9.1.3 Samples with surrogate recovery failures must be re-extracted to confirm a matrix effect. Surrogate recovery failures in method blanks and LCS require re-extraction of the entire batch. Similar surrogate failures in the MS/MSD and original sample confirm a matrix effect and do not require re-extraction.

9.2 Internal Standards

9.2.1 The integrated areas of the quantitation ions of the internal standards are monitored in continuing calibration verification checks, samples, and QC for a change in retention time and response or sensitivity. These should remain reasonably constant over time.

Internal standard retention time and area responses must be assessed in each continuing calibration verification standard by comparison to the corresponding internal standard in the most recent initial calibration mid-point standard. Internal standard responses in samples and QC are compared to the most recent continuing calibration verification.

9.2.2 The area responses of the internal standards must be within 50-200% difference of the area responses compared to.

9.2.3 The retention time for the internal standards must be less than 30 seconds.

9.2.4 If any of these criteria cannot be met, the analytical system must be checked for malfunctions and corrections made. Re-analysis of any affected sample is required.

9.3 Method/Instrument Blanks

9.3.1 Before any samples are analyzed, it must be demonstrated that a laboratory reagent blank is free from contamination that would prevent the determination of any analyte of concern. Sources of background contamination are glassware, purge gas, sorbents, and equipment. Background contamination must be reduced to an acceptable level before proceeding with the next analysis.

9.3.2 All blanks must be analyzed on a GC/MS system meeting the BFB, initial calibration, and continuing calibration verification acceptance criteria.

9.3.3 A method blank is analyzed with each batch of up to 20 samples processed as a group within a 12-hour tune. If more than 20 samples are analyzed in a tune batch, a second method blank is required. Method blanks must be analyzed immediately following a valid continuing calibration verification analysis.

9.3.4 The concentration of the target compounds in the blank must be less than the reporting limit for each target compound, with the exception of the common laboratory contaminants acetone, 2-butanone, and methylene chloride, which must be <2X the reporting limit.

9.3.4.1 The DOD-QSM requires that the target compounds in the blank must be at a concentration of <½ the reporting limit and < the reporting limit for lab contaminants.

9.3.4.2 The SC DHEC requires that all compounds in the blank be less that the reporting limits except for the **common laboratory contaminants**, which must be <2X the reporting limit.

9.3.5 All samples processed within the same 12-hour tune associated with a method blank that does not meet the blank technical acceptance criteria must be reanalyzed.

9.3.6 Method interferences caused by contaminants in solvents, reagents, glassware, laboratory air, and other sample storage and processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms must be eliminated. The chromatographic system must be inspected for malfunctions, and corrections must be made as required

before more samples are analyzed. An instrument blank is analyzed after a high concentration sample in order to eliminate carryover.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS) is prepared and analyzed with each tune batch of up to 20 samples. The LCS and matrix spikes are spiked with the same target analytes. The LCS is spiked at the same concentration as the matrix spike. The LCS is spiked with all compounds of interest in the project.

9.4.2 The percent recovery criteria, developed from in-house statistical data, for the analytes in the full LCS are listed in Attachment 2.

9.4.2.1 The LCS control limits required by the DOD-QSM are listed in Attachment 6 along with the marginal exceedances.

9.4.2.2 The following table presents the DOD-QSM and 2003 NELAC Standards allowed number of marginal exceedances governed by the number of compounds spiked into the LCS.

Number of analytes in the LCS	Allowed number of Marginal Exceedances
> 90	5
71-90	4
51-70	3
31-50	2
11-30	1
< 11	0

The LCS fails if the more than the allowed number of marginal exceedances occur or if a spike recovery is outside of the marginal exceedance limits.

9.4.3 Gases and known poor purging compounds

- gases: bromomethane
  chloromethane
  chloroethane
  vinyl chloride
  dichlorodifluoromethane
  trichlorofluoromethane)
- acetone

- 2-butanone
- carbon disulfide
- crotonaldehyde
- 1,2-dibromo-3-chloropropane
- 1,4-dioxane
- isobutyl alcohol
- 2-hexanone
- 4-methyl-2-pentanone
- vinyl acetate
- 2-chloroethyl vinyl ether

9.4.4 For SC DHEC, an expanded subset of analytes, representative of the compounds being reported, is employed and all analytes must have recovery limits within 70 - 130%. The analytes are:

- vinyl chloride
- 1,1-dichloroethene
- methylene chloride
- 1,1-dichloroethane
- cis-1,2-dichloroethene
- 2-butanone
- carbon tetrachloride
- benzene
- trichloroethene
- 1,2-dichloropropane
- bromodichloromethane
- tetrachloroethene
- chlorobenzene
- ethylbenzene
- styrene
- bromoform
- 1,4-dichlorobenzene

9.4.5 When the LCS fails to meet the acceptance criteria, the entire batch associated with it must be re-prepared and reanalyzed.

9.5 Matrix Spikes

9.5.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared and analyzed with every SDG. The MS/MSD and LCS are spiked with the same analytes.

9.5.2 For the MS/MSD, in addition to spiking internal standard solution and surrogate solution, also add 5.0 µL of 8260 spike solution. For a full LCS requirement, use the ICV standard. The same ICV standard can be used for projects submitted to meet the regulatory requirements of the State of South Carolina, but only the 17 analytes presented in Section 9.4.4 are assessed. The spiking solutions are added by piercing the septum with the syringe needle.

9.5.3 Matrix spikes have the following advisory recovery criteria:

Spike Compound	% Recovery Range
1,1-dichloroethene	59-172
Trichloroethene	62-137
Benzene	66-142
Toluene	59-139
Chlorobenzene	78-122
All others	50-150

9.5.4 Matrix spikes have the following advisory RPD criteria:

Spike Compound	% RPD
1,1-dichloroethene	22
Trichloroethene	24
Benzene	21
Toluene	21
Chlorobenzene	21
All others	25

Note: This table is subject to change without notice.

9.5.5 Most spike compounds should meet these criteria. If the criteria are not met in the MS/MSD but are met in the LCS, the results may be reported with the failures attributed to the matrix of the sample. If the LCS does not meet criteria, then all will have to be repeated as discussed above.

9.5.6 To meet the requirements of the **DoD**-QSM, the duplicate matrix spikes should meet the LCS control limits listed in Attachment 6. The RPD between the duplicate matrix spikes should be ≤ 30%.

9.5.6.1 If the duplicate matrix spikes fail **DoD**-QSM acceptance criteria, notify the project manager. The project manager will contact the client for guidance.

9.5.6.2 If the original sample results are associated with failing duplicate matrix spikes, qualify the results in the narrative as estimated concentrations. Refer to the **DoD**-QSM "J" flag.

9.6 Initial Calibration Verification

9.6.1 A second source initial calibration verification (ICV) standard is run after the initial calibration standards have met criteria.

9.6.2 The ICV must be within 20% of its expected value for each target analyte and surrogate or within 40% for the poor purgers and the gases. Sporadic failure of up to three target compounds is allowed but they must not exceed 40% of their expected value. Gases and poor purgers are listed above.

9.6.2.1 To meet the requirements for the **DoD**-QSM, the ICV must be ± 25% of its expected value for each target analyte.

9.6.3 If the ICV fails to meet the criteria in **Sections** 9.6.2 or 9.6.2.1, take corrective action and reanalyze the standard. If the ICV fails again, repeat the initial calibration.

9.7 MDL Studies

9.7.1 On an annual basis and after major maintenance, a method detection limit (MDL) study is performed on at least one instrument per method and matrix. When multiple instruments are used, individual instrument MDL studies may be replaced by the analysis of an MDL check sample. The MDL check sample must be analyzed on all instruments, to demonstrate equivalent sensitivity.

9.7.1.1 The **DoD**-QSM requires that the MDL check sample be prepared at approximately 2x the MDL and is analyzed on a quarterly basis for each matrix. A response must be detected in the 2x MDL

check sample. Additionally, qualifying ions of 50% or higher must also be present. For more information on MDL studies, refer to QC SOP 13.11, "Performing Annual Method Detection Limit (MDL) Studies."

9.8 Corrective action: Instrument Problems/Preventative Maintenance

9.8.1 If a low response is observed for the early eluting compounds such as the gases, replacement of the trap or septum may be necessary. In addition, adjustments to the purge flow may be necessary to achieve a desired response for these compounds. If such adjustments do not help, it may be necessary to check the fittings on the purge and trap device and on the column for leaks. This is done with a helium leak detector and software utility programs.

9.8.2 Column maintenance or replacement may be necessary if peak tailing or broad chromatographic peaks are observed.

9.9 Contingency

9.9.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.9.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.9.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.9.4 Any other issues that potentially effect data quality must be addressed with the Project Manager.

10.0 <u>Calibration and Standardization</u>

Note: The laboratory reporting limit is at or above the lowest initial calibration standard concentration.

10.1 BFB Tuning

10.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ηg of BFB (2 µL STD ID#7008) into the GC using a 10 µL Hamilton syringe. BFB may be analyzed simultaneously with a continuing calibration verification standard as long as all QC criteria are met.

10.1.2 The peak selection criteria for BFB analysis are as follows (in order of performance):

10.1.2.1 Average one scan prior to the apex of the BFB peak to one scan after the apex, subtracting a single background scan prior to the peak, but no more than 20 scans prior to the elution of BFB. Also, do not subtract part of the BFB peak.

10.1.2.2 Choose the apex of the BFB peak only and include background subtraction.

Note: Background subtraction is performed to eliminate interference and when performed, the subtracted scan must be no more than 20 scans prior to the elution of the BFB and no scans within the BFB peak may be subtracted.

10.1.2.3 Choose a single scan or a range of scans within the BFB peak and include background subtraction.

10.1.3 The analysis of the instrument performance check solution must meet the ion abundance criteria given in the following table.

BFB Key Ions and Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15-40% of m/z 95
75	30-60% of m/z 95
95	Base Peak; 100% relative abundance
96	5-9% of m/z 95
173	<2% of m/z 174
174	>50% of m/z 95

Mass	Ion Abundance Criteria
175	5-9% of m/z 174
176	>95% but less than 101% of m/z 174
177	5-9% of m/z 176

10.1.4 BFB technical acceptance criteria must be met before any standards, samples, or required blanks are analyzed. GC/MS tuning and Mass Calibration forms must be printed and attached to the instrument run log page for each tune. The relative abundance for each ion is calculated to two decimal places.

10.1.5 If BFB technical acceptance criteria are not met, retune the GC/MS system. It may also be necessary to clean the ion source, clean the quadrupole rods, if serviceable, or take other corrective action to achieve the technical acceptance criteria.

10.2 Initial Calibration

10.2.1 Prior to the analysis of samples and required blanks, and after the instrument performance check solution (BFB) criteria have been met, each GC/MS system must be calibrated at six concentrations to demonstrate instrument sensitivity and the linearity of responses for the purgeable target compounds.

10.2.2 Prepare standards according to the Initial Calibration Standard Preparation Table in Section 7.5. The purge and trap volume is 5 mL. All initial calibration standards must be analyzed at the concentration levels and frequency described in this SOP on a GC/MS system meeting the BFB technical acceptance criteria. The analysis of the six calibration standards determines the linearity of the six-point initial calibration curve.

10.2.3 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard. Relative response factors (RRF) are calculated for each compound.

10.2.4 Minimum relative response factors for the System Performance Check Compounds (SPCC) must be met and are listed in the following table.

Relative Response Factor Criteria for SPCCs:

Volatile Compound	**Minimum RRF**
Chloromethane	0.10
1,1-dichloroethane	0.10
Chlorobenzene	0.30
Bromoform	0.10
1,1,2,2-tetrachloroethane	0.30

10.2.5 The following Calibration Check Compounds (CCC) have maximum % RSD criteria of ≤ 30%:

- 1,1-dichloroethene
- chloroform
- 1,2-dichloropropane
- toluene
- ethylbenzene
- vinyl chloride

10.2.5.1 The remaining compounds must have an RSD of ≤ 15%. If the % RSD is 15% or less, the average relative response factor may be used for quantitation. If the % RSD is greater than 15% then an alternate method for quantitation, such as a linear calibration using least squares regression or quadratic fit, may be used. When one of these options is used, the line must not be forced through the origin.

If the linear or quadratic regression fit is used, the correlation coefficient must be ≥ 0.990. These alternate methods of quantitation are available in the ThruPut system.

Note: The **DoD**-QSM requires that the linear least squares regression correlation coefficient must be ≥ 0.995 and does not allow the used of quadratic fit.

10.2.5.2 If the initial calibration does not meet the criteria above, corrective action is necessary.

- Check the instrument operating conditions and perform maintenance as necessary. It may be necessary to clean the ion source, perform column maintenance, change the column, service the purge and trap device, or take other

corrective action to achieve the technical acceptance criteria.

- Compare responses for the analyte in each of the standard levels to verify that a single standard analysis is not presenting results significantly higher or lower then the other standard analyses, as this would indicate that the standard solution was prepared in error. If that is the case, re-prepare and reanalyze the standard.

10.2.7 The initial calibration verification must be analyzed after each initial calibration and must meet the acceptance criteria. If the ICV fails, then a new initial calibration curve must be generated.

10.3 Continuing Calibration Verification

10.3.1 Before the analysis of samples and blanks, but after BFB and initial calibration acceptance criteria have been met, each GC/MS system must be routinely checked by analyzing a continuing calibration verification standard. This standard contains all purgeable target analytes and surrogate compounds. It is used to ensure that the instrument meets the sensitivity and linearity requirements of the method throughout the analytical sequence.

10.3.2 A check of the calibration curve must be performed once every 12 hours, beginning with the injection of BFB. A percent difference of the response for each compound compared to the mean relative response factor from the initial calibration is calculated when performing the average response factor model.

10.3.3 The calculated percent difference must be less than or equal to 20% for the CCCs listed above in Section 10.2.5. Minimum response factor criteria for the continuing calibration verification standard (SPCCs) are also shown in Section 10.2.4.

10.3.3.1 If a regression fit model was used for analytes in the initial calibration, the continuing calibration verification is performed using percent drift (difference) for the CCCs.

10.3.4 As indicated for the initial calibration acceptance criteria, for the continuing calibration verification, the remaining target analytes (non-CCC compounds) do not have defined % difference criteria. We have

established a warning limit of 50%D and an action limit of 90%D. These values have been inserted as defaults into the data reduction software program. This is based strictly on established U.S. EPA data validation guidelines where values greater than 90% RSD results in rejection of data.

10.3.4.1 For samples submitted to meet the regulatory requirements of the State of South Carolina, all non-CCC target analytes must be within 30% acceptance criteria, with the exception of the poor purgers listed in Section 9.4.3. The poor purgers should fall with 40% acceptance criteria.

10.3.5 If continuing calibration verification acceptance criteria cannot be met after inspection and normal maintenance, a new initial calibration will have to be performed.

Note: Method 8260B indicates that if the CCCs are not required analytes, then all required analytes, must meet the 20% difference/drift criterion. Our typical analysis includes all of the CCCs.

11.0 Procedure

Documentation must follow the requirements in QC SOP 13.6: "Proper Documentation Procedures". All injections must be recorded on the instrument runlog (Attachment 4) along with the date, time (use a 24 hour clock), the volume injected, operator ID, and any comments relevant to the injection. Circle the Method used to perform the analysis. Condition codes, abbreviations defined in Attachment 8, are used to document failed and qualified injections. Preventive maintenance such as clipping the front-end of the column must be recorded in the logbook. Any major maintenance, i.e., changing the source or column, must be recorded in the instrument maintenance logbook.

All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.1 Instrument Software Conventions

11.1.1 Quantitation method: Average of the whole

11.1.2 File naming convention: XX123456X78

where: XX = Analysis type
123456 = Date/Lab ID with first digit dropped
Y = shift
78 = instrument #

11.1.3 Analysis type prefixes

11.1.3.1 Standard: CS, CT, CU, CV, CW, CX

11.1.3.2 Initial Sample Injection:

- SDG-Sample Number-Shift-Instrument, e.g., Q1636-1A52

11.1.3.3 Sample reinjection:

- SDG-Sample Number-J-Shift-Instrument, e.g., Q1636-1JA52

11.1.3.4 Sample re-extraction:

- SDG-Sample Number-R-Shift-Instrument, e.g., Q1636-1RA52

11.1.3.5 Sample dilution:

- SDG-Sample Number-D-Shift-Instrument, e.g., Q1636-1DA60

11.1.3.6 Additional repeats:

- SDG-Sample Number-(J2, R2, D2)-Shift-Instrument, e.g., Q1636-1J2A52

11.2 Analytical Sequence

11.2.1 Order of analysis for the instrument calibration:

- BFB (tune)
- initial calibration
- initial calibration verification

11.2.2 Order of analysis for the twelve-hour tune:

- BFB
- continuing calibration verification
- instrument blank
- laboratory control sample-LCS
- samples

11.2.3 All samples must be analyzed on a GC/MS system meeting the BFB, initial calibration, continuing calibration verification, and instrument blank criteria.

11.2.3.1 If tune time remains after the initial calibration standards have been run, samples may be analyzed as long as they are preceded by a valid instrument blank.

11.3 Preparations

11.3.1 Standards

11.3.1.1 The analysis of the instrument performance check solution is performed by injecting 50 ng of BFB (2 µL Standard ID#7008) into the GC using a 10 µL Hamilton syringe.

11.3.1.2 Calibration standards are prepared by spiking the appropriate volume of each standard solution into 5 mL of sparged DI water contained in a 5 mL syringe. This is then added to a 40 mL vial which is immediately capped with a PTFE-lined, septum-sealed cap and loaded into the Archon autosampler.

Initiate the Archon autosampler which will provide mixing, the addition of 5 mL of water containing 1 µL of internal standards and 1 µL of surrogate (5 µL of 50 µg/mL solutions if spiked by the chemist and not the Archon), heating at 40 °C, and purging for 11 minutes. The system will then transfer the constituents in the headspace to the Tekmar 3000 purge and trap concentrator and will then desorb all target analytes for 4 minutes before analysis. The analyses of the six calibration standards determine the linearity of the six-point initial calibration curve.

11.3.2 Instrument Blank and Method Blank

11.3.2.1 An instrument blank is prepared by filling a 40 mL VOA vial with 5 mL of purged DI water and sealing with a screw-top, PFTE-faced, septum-sealed cap. This is placed into the Archon autosampler where DI water, 1 µL of internal standards, and 1 µL of surrogates are added automatically to the blank (5 µL of 50 µg/mL solutions if spiked by the chemist and not the Archon). It is analyzed by a closed system heated purge and trap analysis.

11.3.2.2 A Method Blank is similar to an Instrument Blank in composition but it is prepared at the same time samples are prepared and is stored in the refrigerator. If samples are received from the field already in vials with sodium bisulfate preservative solution, only an Instrument Blank is required.

11.3.3 Laboratory Control Sample

11.3.3.1 A laboratory control sample (LCS) is prepared by filling a 40 mL VOA vial with 5 mL of purged DI water. To this 10 µL of the spiking standard is added before sealing with a screw-top, PFTE-faced, septum-sealed cap. This is placed into the Archon autosampler where DI water, 1 µL of internal standards, and 1 µL of surrogates are added automatically (5 µL of 50 µg/mL solutions if spiked by the chemist and not the Archon). It is analyzed by a closed system heated purge and trap analysis.

11.3.3.2 For certain projects and programs, a full list spike is required.

11.3.4 Samples

11.3.4.1 Solid samples are prepared by Method 5035. For details see Sample Preparation Procedure –238: "**Preparation of Soil / Sediment / Sludge / Non-aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap by SW-846 Methods 5035A and 3585, and EPA CLP SOW OLM04.3**."

11.3.4.2 The choice of whether a 5 g or medium level sample is analyzed is generally based on a screen analysis.

11.3.4.3 Samples are stored in a rack located in the volatile GC/MS freezer at -10 to -20 °C. Samples are allowed to come to room temperature and, 5 mL of DI water added by piercing the septum, then loaded into the Archon autosampler carousel shaking each vial gently so that the contents move freely.

11.3.4.4 In some case, samples are preserved with 5 mL of a solution of sodium bisulfate. In those cases, the vials are removed from a refrigerator, maintained at 2-6 °C, allowed to come to room temperature and loaded into the Archon autosampler, shaking

each vial gently so that the contents move freely and the stirring bar will be able to spin.

11.3.5 Matrix Spikes

11.3.5.1 For sample spikes, the Archon will spike 1 µL of internal standard solution and 1 µL of surrogate solution (5 µL of 50 µg/mL solutions is spiked by the chemist and not the Archon), also add 10.0 µL of 8260B spike solution.

11.3.5.2 For certain projects, full target list matrix spikes are required.

11.4 Analysis

11.4.1 When the Archon autosampler is initiated, the system will add 5 mL of purged DI water, containing 5 µL of internal standards and 5 µL of surrogates, by piercing the septum.

11.4.2 Prior to purging, the sample is heated to 40 °C. The sample is purged for 11 minutes, while mixing. For soil samples preserved with a sodium bisulfate solution, the stirring bar is turned on.

11.4.3 The same needle that is used to add the DI water is the source for the inert gas used for purging. The needle also contains slots above the sample/water level which provide a path for the headspace to be directed to the Tekmar 3000 purge and trap concentrator. This contains the trap that is then thermally desorbed into the GC/MS instrument.

11.4.4 After purging, the Purge and Trap Concentrator apparatus will desorb onto the GC column by elevating the trap temperature to 260 °C and back-flushing the trap with helium for 4 minutes at 20 to 60 mL/minute.

11.4.5 After desorbing, the trap is reconditioned by baking at 260 °C for at least 7 minutes. When the trap has finished baking and is cool, it is ready for the next sample to be purged.

11.4.6 In each analytical run, all analytes must fall below the maximum calibration range established by the highest standard in the initial calibration. If an analyte is present at a concentration higher than the highest initial calibration standard, it must be reanalyzed at a lesser amount or dilution. A valid dilution is one in which the compound in

question falls above the mid-point calibration standard concentration. The dilution is considered valid if the analyte concentration is above 50 µg/kg.

11.5 Target Compound Identification

11.5.1 Target compounds are identified in the samples by analyzing standards under the same conditions used for samples. The resulting mass spectra are compared to established library spectra and GC retention times to retention times from the latest continuing calibration standard. The mass spectrum of the sample compound and a laboratory library-generated spectrum must match according to the following criteria:

11.5.1.1 All ions present in the library mass spectrum at a relative intensity >10% must be present in the sample spectrum.

11.5.1.2 The relative intensities of ions specified above must agree within ± 20% between the library and sample spectra.

11.5.1.3 Ions >10% in the sample spectrum but not present in the library spectrum must be considered and accounted for. (These ions may be a result of co-eluting/closely-eluting compounds.)

11.5.2 If a compound analyzed by GC/MS techniques cannot be verified by all of the criteria listed above, but in the technical judgment of the mass spectral interpretation specialist the identification is correct, then the laboratory will report that identification.

11.5.3 If any of the internal standards, surrogates, or spike compounds are missing or failing, check the peak integration in "Target Review". Changes can be made in Target Review and EICPs can be generated. Any compound for which manual peak integration has been performed will have an "M" flag displayed on the quantitation report. All manual integration must follow the procedures documented in SOP 13.18, "Manual Chromatographic Peak Integration Procedures." These entries must be assigned a numerical code and the analyst's initials and the date must appear on each quantitation report page containing an "M" flag. For multiple "M" flags appearing on a quantitation report page, a bracket encompassing the flags can be used, with a single entry of the initials and date. The department supervisor or a representative of the QA department must approve of all manual integrations performed on all initial and continuing calibration standards. This is documented by initialing and dating each page of the quantitation report of the raw data containing such

manual integrations. See Attachment 9 for definitions of manual integration codes.

11.5.4 Non-target compounds are identified by comparing the resultant mass spectra from the non-target compounds to mass spectra contained in the National Institute of Standards and Technology (NIST) Mass Spectral Library. See Section 11.6.

11.6 Qualitative Analysis of Non-Target Compounds

11.6.1 Tentatively identify all compounds in the sample that have not already been identified as target compounds, or that are not surrogates, internal standards or semivolatile target compounds, by performing a computer-generated library search using the NIST 129K.1 mass spectral library (See Section 6.10.3). The library search must not be normalized.

11.6.2 Up to 30 tentatively identified compounds (TICs), including alkanes, of the greatest concentration are reported for each sample. The number of TICs required may vary, depending on project requirements.

11.6.3 Rules for making tentative identification:

11.6.3.1 TICs receiving a library search match of 85% or higher are considered a "probable match". Report the compound identified.

11.6.3.2 For TICs receiving more than one library search match of 85% or higher, report the compound with the highest percent match.

11.6.3.3 For TICs receiving two or more library search matches of 85% or higher with the same percent match, report the first compound.

11.6.3.4 For TICs that are isomers receiving library search matches of 85% or higher, report the compound with the highest percent match.

Note: If in the opinion the experienced analyst/data reviewer there is sound technical evidence not to identify the compound as specified in Sections 11.6.3.1 to 11.6.3.4 the justification must be documented in the narrative.

11.6.3.5 TICs receiving a library search match of < 85% are given a tentative identification by the analyst/data reviewer, if possible. If no identification can be made the TIC is reported as "unknown". If possible the unknown is further identified as part of a class of compounds (for example "unknown aromatic").

11.7 Quantitation

11.7.1 The mean relative response factor (RRF) from the initial calibration standard is used to calculate the concentration in the sample. For NYSASP, the RRF from the continuing calibration standard is used to calculate concentrations.

Note: Alternatively, the calibration curve(s) generated from the initial calibration may be used for the determination of analyte(s) concentration(s). This option is discussed above.

11.7.2 All samples require a search of all extraneous peaks >10% of the height of the nearest internal standard, up to 10 searches, i.e**,**. 10 most intense extraneous peaks. The number of searches may be more, depending on client requirements.

11.7.3 In each analytical run, all analytes must fall below the method's maximum analytical range, i.e. the highest calibration standard.

11.7.3.1 If an analyte is present at a concentration higher than the maximum analytical range in a 5g analysis, the medium level sample must be analyzed.

11.7.4 When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by the analysis of an instrument blank or the following sample must be monitored for contamination and interference from carryover. If the blank or sample is not free from interferences, the system must be decontaminated. Sample analysis may not resume until a blank or sample has been analyzed which is free from interferences. Being free from interferences means that whatever compound was present above the initial calibration range in a sample, cannot be present in an instrument blank or the sample analyzed immediately following, at a level above the reporting limit for that compound.

11.7.5 Secondary ion quantitation is allowed only when there are sample matrix interferences with the primary ion. If secondary ion quantitation is performed, document the reasons in the SDG narrative.

11.7.6 Non-target compounds are quantified by comparing the MS response from the reconstructed ion chromatogram (RIC) for the non-target compound peaks to the MS response for a peak produced by the nearest internal standard compound. A response factor of 1 is assumed.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\,R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of %RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{(Value\ 1\ +\ Value\ 2)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value\ -\ \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax\ x\ C(is)}{A(is)\ x\ Cx}$$

where: Ax = Area of the characteristic ion (EICP) for the compound to be measured
A(is) = Area of the characteristic ion (EICP) for the specific internal standard
C(is) = Concentration of the internal standard (in µg/**L**)
Cx = Concentration of the compound to be measured

12.8 Linear Calibration using Least Squares Regression

General linear equation: $y = ax + b$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Mass of the analyte in the calibration standard aliquot injected
b = y-intercept

Linear Regression by Least Squares:

$$y_1^{'} = ax_i + b$$

Where: b = intercept
x_i = Mass of the analyte in the ith calibration standard aliquot injected
$y_1^{'}$ = calculated response for the ith calibration standard

The sum of the squares of the differences is minimized to obtain a and b:

12.9 Correlation Coefficient r:

$$r = \frac{N\left[\sum_{i=1}^{N}(x_i y_i)\right] - \left[\sum_{i=1}^{N}(x_i)\right]\left[\sum_{i=1}^{N}(y_i)\right]}{\sqrt{N\left[\sum_{i=1}^{N}(x_i^2)\right] - \left[\sum_{i=1}^{N}(x_i)\right]^2} * \sqrt{N\left[\sum_{i=1}^{N}(y_i^2)\right] - \left[\sum_{i=1}^{N}(y_i)\right]^2}}$$

Where:
r = Correlation Coefficient
N = number of data points (equals 5 in a 5 point curve)
y = response
i = index variable (first data point i = 1)
x = Mass of the analyte in the calibration standard aliquot injected

12.10 Quadratic Calibration (second order: $y = ax^2 + bx + c$) fit in Target

$$y = [n]\left[b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)\right]$$

where: b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
x = area of analyte/area of Internal Standard
n = amount of Internal Standard
y = amount in ng on column
Rsp = area of analyte/area of Internal Standard

12.11 Concentration

12.11.1 The area response of the characteristic ions in the extracted ion current profile (EICP) is tabulated against the concentration for each compound and internal standard.

12.11.2 Concentration of soil samples (dry weight basis) by GC/MS analysis using relative response factor:

$$ug/kg = \frac{(As)(Cis)}{(Ais)(\overline{RF})(Ws)(D)}$$

where: As = Area of the peak for the analyte in the sample
Ais = Area of the peak for the internal standard
Cis = Concentration of the internal standard in the volume purged, in µg/L
$\overline{RF}$ = Mean response factor from the initial calibration

$$\overline{RF} = \frac{\sum_{i=1}^{n} RFi}{n}$$

Ws = weight of sample purged, in grams

$$D\ (dry\ weight) = \frac{100 - \%\ moisture}{100}$$

12.11.3 Concentration of soil samples (dry weight basis) by GC/MS using quadratic (second order) fit in Target:

$$y = [n]\left[b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)\right]$$

where:b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
n = amount of Internal Standard
y = concentration in ng on column
Rsp = area of analyte/area of Internal Standard

Example:Area of acetone = 35659
Area of IS = 613275
b = -0.0909161

$m^1 = 9.605304$
$m_2 = 7.132688$
ng of IS = 250
response = 35659/613275 = 0.058145

Amount in ng on column =

$$(250ng)[-0.0909161 + 9.605304 \ x \ 0.058145 + 7.132688 \ x \ 0.058145^2] = 122.9ng$$

$$Concentration \ \mu g / Kg = \frac{122.9ng}{(Ws)(D)}$$

12.11.4 Concentration of soil samples (dry weight basis) by GC/MS using linear regression analysis:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

where: As = Area of the target analyte peak in the sample
Ais = Area of the internal standard peak
Cs = Concentration of the target analyte in the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (coefficient of Cs)
b = The intercept

$$Concentration \ \mu g / Kg = \frac{(Cs)}{(\mathrm{Ws})(D)}$$

12.11.5 Tentatively Identified Compound (TIC) Estimation

$$TIC \ Amount = \frac{(Area \ TIC) \ x \ Amount(Std)}{(Area \ IS) \ x \ 1(RF)(Ws)(D)}$$

where: Area (TIC) = area response from RIC for non-target compound
Amount(Std) = amount of internal standard added to the sample, in µg/L.

Area (IS)	=	area response of the nearest internal standard in the reconstructed ion chromatogram
1(RF)	=	assumed response factor of 1

12.12 Calculating Dilutions

12.12.1 If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution.

12.12.2 Adjust the amount of sample purged with 1 gram being the lowest acceptable weight for a low level analysis. If the analyte still exceeds the analytical range in the medium level analysis, perform a methanol extraction following Sample Preparation Procedure –238, "**Preparation of Soil/Sediment/Sludge/Non-aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap by SW-846 Methods 5035A and 3585, and EPA CLP SOW OLM04.3**".

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste: Physical/Chemical Methods. U.S. EPA SW846 3rd Edition, Update 3, 12/96, Methods 8000C, 8260B and 5035A

16.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 QC SOP 13.6: "Proper Documentation Procedures"

16.5 QC SOP 13.4: "Numerical Data Reduction"

16.6 NELAC Standards, effective July 2003, plus revisions

16.7 QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/027, April 2007.

16.8 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, **10/25/2010**

16.9 CompuChem Quality Manual, Revision ~~13, November 17, 2009,~~ 14, January 25, 2011 plus revisions

16.10 Sample Control SOP 4.1, "Receiving Samples"

16.11 Sample Control SOP 4.6, "Storing Samples"

16.12 QC SOP 13.11, "Performing Annual Method Detection Limit (MDL) Studies"

16.13 Sample Preparation Procedure –238, "**Preparation of Soil / Sediment / Sludge / Non-aqueous Waste Samples for the Analysis of Volatile Organic Compounds by Closed-System Purge and Trap by SW-846 Methods 5035A and 3585, and EPA CLP SOW OLM04.3**"

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 – Target Analyte List

17.2 Attachment 2 – In-house Statistical Control Limits

17.3 Attachment 3 – Target Analyte Reporting Limits

17.4 Attachment 4 – **Example** Instrument Run Log

17.5 Attachment 5 – Initial Calibration Standard Mixes

17.6 Attachment 6 – **DoD**-QSM LCS Control Limits

17.7 Attachment 7 – Second Source Initial Calibration Verification Standard Mixes

17.8 Attachment 8 – Definitions of Condition Codes

17.9 Attachment 9 – Definitions of Manual Integration Codes

Attachment 1

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
dichlorodifluoromethane	1	85	87
chloromethane	1	50	52
vinyl chloride	1	62	64
bromomethane	1	94	96
chloroethane	1	64	66
trichlorofluoromethane	1	101	103
1,1-dichloroethene	1	96	61, 98
methylene chloride	1	84	49, 86
trans-1,2-dichloroethene	1	96	61, 98
1,1-dichloroethane	1	63	65, 83
2,2-dichloropropane	1	77	97
cis-1,2-dichloroethene	1	96	61, 98
bromochloromethane	1	128	49, 130
chloroform	1	83	85
1,1,1-trichloroethane	1	97	99, 61
carbon tetrachloride	1	117	119, 121
1,1-dichloropropene	1	75	110, 77
benzene	1	78	77, 51
1,2-dichloroethane	1	62	98
trichloroethene	1	130	95, 97
1,2-dichloropropane	1	63	112
dibromomethane	1	174	93, 95
bromodichloromethane	1	83	85, 127
2-chloroethyl vinyl ether	1	63	65, 106
cis-1,3-dichloropropene	1	75	77
acrolein	1	56	55, 58
iodomethane	1	142	127, 141
1,1,1-trichloro-2,2,2,-trifluoroethane	1	117	151, 153
1,1,2-trichloro-1,2,2,-trifluoroethane	1	85	101, 151
carbon disulfide	1	76	78
acetone	1	43	58
3-chloropropene	1	76	41, 78
acetonitrile	1	41	40, 39

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Attachment 1 (continued)

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
Acrylonitrile	1	53	52, 51
Methyl-tert-butyl ether	1	73	41, 43
Vinyl acetate	1	43	86
2-Butanone	1	72	43, 57
Propionitrile	1	54	55, 52
Methacrylonitrile	1	41	39, 67
1-Chlorobutane	1	56	49
1,4-Dioxane	1	88	58
Methylmethacrylate	1	69	100, 41
Dibromofluoromethane: Surrogate #1	1	113	111, 192
1,2-Dichloroethane-d_4: Surrogate #2	1	65	102, 67
4-Methyl-2-pentanone	2	43	85, 100
Toluene	2	92	91
trans-1,3-Dichloropropene	2	75	77
1,1,2-Trichloroethane	2	97	83, 85
Ethylmethacrylate	2	69	41, 99
Tetrachloroethene	2	164	168, 129
1,3-Dichloropropane	2	76	78
2-Hexanone	2	43	58, 57
Dibromochloromethane	2	129	127, 48
1,2-Dibromoethane	2	107	109, 188
Chlorobenzene	2	112	114, 77
1,1,1,2-Tetrachloroethane	2	131	119, 133
Ethylbenzene	2	106	91
m,p-Xylene	2	106	91
o-Xylene	2	106	91
Styrene	2	104	91, 78
Bromoform	2	173	175, 254
Isopropyl benzene	2	105	120
Bromobenzene	2	156	77,158
1,1,2,2-Tetrachloroethane	2	83	85, 131

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Attachment 1 (continued)

Volatile Target Compounds

Compounds	Internal Standard	Primary Quantitation Ion	Secondary Quantitation Ion(s)
1,2,3-Trichloropropane	2	110	75, 112
trans-1,4-Dichloro-2-butene	2	53	88, 75
Toluene-d_8: Surrogate #3	2	98	70, 100
n-Propyl benzene	3	91	120
2-Chlorotoluene	3	126	91
4-Chlorotoluene	3	91	126
1,2,4-Trimethyl benzene	3	105	120
1,3,5-Trimethyl benzene	3	105	120
Pentachloroethane	3	167	130, 165
sec-Butyl benzene	3	105	134
1,2-Dichlorobenzene	3	146	111, 148
1,3-Dichlorobenzene	3	146	111, 148
1,4-Dichlorobenzene	3	146	111, 148
n-Butyl benzene	3	91	92, 134
tert-Butyl benzene	3	119	91, 134
p-Isopropyl toluene	3	119	134, 91
1,2-Dibromo-3-chloropropane	3	75	155, 157
1,2,4-Trichlorobenzene	3	180	182, 145
Hexachlorobutadiene	3	225	223, 227
Naphthalene	3	128	64, 51
1,2,3-Trichlorobenzene	3	180	182, 145
4-Bromofluorobenzene: Surrogate #4	3	95	174, 176
Fluorobenzene: Internal Standard #1	NA	96	70
Chlorobenzene-d_5: Internal Standard #2	NA	117	82, 119
1,4-Dichlorobenzene-d_4:Internal Standard #3	NA	152	150

[1] Based on laboratory tests, 2-chloroethyl vinyl ether is not analyzable from the sodium bisulfate solution associated with Method 5035.

Attachment 2

Statistical Control Limits for the LCS

Compound	Percent Recovery Range 5 gram soil
Dichlorodifluoromethane	50-142
Chloromethane	52-150
Vinyl chloride [2]	58-148
Bromomethane	50-150
Chloroethane	52-150
Trichlorofluoromethane	54-144
Acrolein	50-150
1,1-Dichloroethene [1, 2]	51-146
Iodomethane	50-150
1,1,1-Trichloro-2,2,2,-trifluoroethane	58-148
Carbon disulfide	56-140
1,1,2-Trichloro-1,2,2,-trifluoroethane	50-149
Acetone	50-147
3-Chloropropene	50-150
Acetonitrile	68-126
Methyl acetate	50-150
Methylene chloride [2]	50-137
trans-1,2-dichloroethene	65-122
Acrylonitrile	50-136
Methyl-tert-butyl ether	70-133
Tert butyl alcohol	60-148
n-Hexane	50-143
1,1-Dichloroethane [2]	66-123
Chloroprene	50-150
Vinyl acetate	50-150
Isopropyl ether	81-114
2,2-Dichloropropane	64-141
cis-1,2-Dichloroethene [2]	69-127
2-Butanone [2]	66-132
Propionitrile	50-150
Bromochloromethane	73-126
Methyl acrylate	50-133
Methacrylonitrile	51-127
Tetrahydrofuran	50-150

Attachment 2 (continued)

Statistical Control Limits for the LCS

Compound	Percent Recovery Range 5 gram soil
Chloroform	75-128
1,1,1-trichloroethane	71-127
Cyclohexane	65-126
Carbon tetrachloride [2]	67-132
1,1-Dichloropropene	72-132
Benzene [1, 2]	67-126
1,2-Dichloroethane	56-141
Isobutyl alcohol	50-150
Trichloroethene [1, 2]	69-130
Methylcyclohexane	80-119
1,2-Dichloropropane [2]	73-121
Dibromomethane	64-131
1,4-Dioxane	50-150
Methylmethacrylate	60-127
Bromodichloromethane [2]	75-133
2-Chloroethyl vinyl ether	50-150
cis-1,3-Dichloropropene	87-127
4-Methyl-2-pentanone	67-125
Toluene [1]	73-121
trans-1,3-Dichloropropene	64-131
1,1,2-Trichloroethane	67-123
Ethylmethacrylate	67-124
Tetrachloroethene [2]	72-130
1,3-Dichloropropane	75-130
2-Hexanone	51-128
Dibromochloromethane	68-127
1,2-Dibromoethane	69-127
Chlorobenzene [1, 2]	71-120
1-Chlorohexane	80-113
1,1,1,2-Tetrachloroethane	82-120
Ethylbenzene [2]	76-121
m,p-Xylene	76-130

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Attachment 2 (continued)

Statistical Control Limits for the LCS

Compounds	Percent Recovery Range 5 gram soil
o-Xylene	76-130
Styrene [2]	68-126
Bromoform [2]	64-134
Isopropyl benzene	68-126
Bromobenzene	67-140
1,2,3-Trichloropropane	58-137
1,1,2,2-Tetrachloroethane	66-128
trans-1,4-Dichloro-2-butene	50-150
n-Propylbenzene	71-136
2-Chlorotoluene	78-124
4-Chlorotoluene	74-131
1,3,5-Trimethyl benzene	72-129
Pentachloroethane	64-150
tert-Butyl benzene	67-140
1,2,4-Trimethyl benzene	70-133
sec-Butyl benzene	67-141
1,3-Dichlorobenzene	74-119
1,4-Dichlorobenzene [2]	72-115
p-Isopropyl toluene	74-132
1,2-Dichlorobenzene	67-126
n-Butyl benzene	63-137
1,2-Diethylbenzene	75-117
1,2-Dibromo-3-chloropropane	72-127
1,2,4-Trichlorobenzene	57-133
Hexachlorobutadiene	68-129
Naphthalene	50-139
1,2,3-Trichlorobenzene	63-129
Xylene (total)	76-130

Table displays statistical control limits calculated in 2002.

[1] Denotes component of minimum LCS spike
[2] Denotes component of LCS spike for SC DHEC. Each of these analytes must be recovered within 70 - 130%.

Attachment 3

8260B Compounds and Reporting Limits

CAS#	Compound	5 grams (µg/Kg)
630-20-6	1,1,1,2-Tetrachloroethane	5
354-58-5	1,1,1-Trichloro-2,2,2-trifluoroethane	5
71-55-6	1,1,1,-Trichloroethane	5
79-34-5	1,1,2,2-Tetrachloroethane	5
76-13-1	1,1,2-Trichloro-1,2,2-trifluoroethane	5
79-00-5	1,1,2-Trichloroethane	5
75-34-3	1,1-Dichloroethane	5
75-35-4	1,1-Dichloroethene	5
563-58-6	1,1-Dichloropropene	5
544-10-5	1-Chlorobenzene	5
87-61-6	1,2,3-Trichlorobenzene	5
96-18-4	1,2,3-Trichloropropane	5
120-82-1	1,2,4-Trichlorobenzene	5
95-63-6	1,2,4-Trimethylbenzene	5
96-12-8	1,2-Dibromo-3-Chloropropane	5
106-93-4	1,2-Dibromoethane	5
95-50-1	1,2-Dichlorobenzene	5
107-06-2	1,2-Dichloroethane	5
78-87-5	1,2-Dichloropropane	5
108-67-8	1,3,5-Trimethylbenzene	5
541-73-1	1,3-Dichlorobenzene	5
142-28-9	1,3-Dichloropropane	5
106-46-7	1,4-Dichlorobenzene	5
123-91-1	1,4-Dioxane	250
594-20-7	2,2-Dichloropropane	5
78-93-3	2-Butanone	12.5
110-75-8	2-Chloroethyl vinyl ether	5
95-49-8	2-Chlorotoluene	5
591-78-6	2-Hexanone	12.5
107-05-1	3-Chloropropene	5
106-43-4	4-Chlorotoluene	5
108-10-1	4-Methyl-2-pentanone	12.5
67-64-1	Acetone	12.5
75-05-8	Acetonitrile	5
107-02-8	Acrolein	50

Attachment 3 (continued)

8260B Compounds and Reporting Limits

CAS#	Compound	5 grams µg/Kg
107-13-1	Acrylonitrile	50
71-43-2	Benzene	5
108-86-1	Bromobenzene	5
74-97-5	Bromochloromethane	5
75-27-4	Bromodichloromethane	5
75-25-2	Bromoform	5
74-83-9	Bromomethane	5
75-15-0	Carbon disulfide	5
56-23-5	Carbon Tetrachloride	5
108-90-7	Chlorobenzene	5
75-00-3	Chloroethane	5
67-66-3	Chloroform	5
74-87-3	Chloromethane	5
126-99-8	Chloroprene	5
156-59-2	cis-1,2-Dichloroethene	5
10061-01-5	cis-1,3-Dichloropropene	5
110-82-7	Cyclohexane	5
124-48-1	Dibromochloromethane	5
74-95-3	Dibromomethane	5
75-71-8	Dichlorodifluoromethane	5
100-41-4	Ethylbenzene	5
97-63-2	Ethylmethacrylate	50
87-68-3	Hexachlorobutadiene	5
74-88-4	Iodomethane	5
78-83-1	Isobutyl alcohol	250
89-82-8	Isopropyl Benzene	5
108-20-3	Isopropyl ether	5
108-38-3	m,p-Xylene	10
126-89-7	Methacrylonitrile	50
79-20-9	Methyl acetate	5
108-87-2	Methylcyclohexane	5
75-09-2	Methylene Chloride	5
80-62-6	Methylmethacrylate	50
1634-04-4	Methyl-tert-butyl ether	5
91-20-3	Naphthalene	5
104-51-8	n-Butyl benzene	5
110-54-3	n-Hexane	5

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Attachment 3 (continued)

8260B Compounds and Reporting Limits

CAS#	Compound	5 grams µg/Kg
103-65-1	n-Propyl benzene	5
95-47-6	o-Xylene	5
76-01-7	Pentachloroethane	5
99-87-6	p-Isopropyl Toluene	5
107-12-0	Propionitrile	250
135-98-8	sec-Butyl benzene	5
100-42-5	Styrene	5
75-65-0	Tert-Butyl alcohol	5
98-06-6	tert-Butyl benzene	5
127-18-4	Tetrachloroethene	5
109-99-9	Tetrahydrofuran	20
108-88-3	Toluene	5
156-60-5	trans-1,2-Dichloroethene	5
10061-02-6	trans-1,3-Dichloropropene	5
110-57-6	trans-1,4-Dichloro-2-butene	20
79-01-6	Trichloroethene	5
75-69-4	Trichlorofluoromethane	5
108-05-4	Vinyl acetate	5
75-01-4	Vinyl Chloride	5
1330-20-7	Xylene (total)	15

Attachment 4

COMPUCHEM a Division of Liberty Analytical Corp DATE ___/___/___ INITIAL TIME OF TUNE_____ SHIFT/S(A)_____(B)_____(C)____
GC/MS VOLATILE RUN LOG TIME TUNE EXPIRES_____ LINKER /METHOD_____
COMPUCHEM LOGBOOK 11 NNN 29 (5972hp59) METHOD: 8260B SOM01.2 (Circle one) or Other: _____
MAINTENANCE_____

	REJECTED	FILE NAME	REPORTED	pH	Vial	DATE	TIME	Client ID	SDG#	INJ. VOL.*	DF+	CHEMIST	COMMENTS (ETC.)/ DISPOSITION
1						/ /							
2						/ /							
3						/ /							
4						/ /							
5						/ /							
6						/ /							
7						/ /							
8						/ /							
9						/ /							
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22						/ /							
23						/ /							
24						/ /							

*On-column sample aliquot +Not applicable to soil matrix

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation

Tune (ID #7008) Lot No.: _____ Calibration Group Code / Lot No.: _____
Internal Standard Lot No.: _____ Surrogate Lot No.: _____

REVIEWED BY: _____ Date_____

4/05/10: jad

Note: This page subject to change without notice.

Attachment 5

TCL4 1&2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

Cat. No.: 30431 Lot No.: A027506
Description: 502.2 CAL 2000 Mega-Mix
Expiration Date[1]: August 2005 Storage: Freezer

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1,1-dichloroethane	75-34-3	99%	2,000 µg/ml	+/- .04%
1,1-dichloroethene	75-35-4	99%	2,000 µg/ml	+/- .04%
1,1-dichloropropene	563-58-6	99%	2,000 µg/ml	+/- .04%
1,1,1-trichloroethane	71-55-6	99%	2,000 µg/ml	+/- .04%
1,1,1,2-tetrachloroethane	630-20-6	99%	2,000 µg/ml	+/- .04%
1,1,2-trichloroethane	79-00-5	99%	2,000 µg/ml	+/- .04%
1,1,2,2-tetrachloroethane	79-34-5	99%	2,000 µg/ml	+/- .04%
1,2-dibromo-3-chloropropane	96-12-8	97%	2,000 µg/ml	+/- .04%
1,2-dibromoethane	106-93-4	99%	2,000 µg/ml	+/- .04%
1,2-dichlorobenzene	95-50-1	99%	2,000 µg/ml	+/- .04%
1,2-dichloroethane	107-06-2	99%	2,000 µg/ml	+/- .04%
1,2-dichloropropane	78-87-5	99%	2,000 µg/ml	+/- .04%
1,2,3-trichlorobenzene	87-61-6	99%	2,000 µg/ml	+/- .04%
1,2,3-trichloropropane	96-18-4	99%	2,000 µg/ml	+/- .04%
1,2,4-trichlorobenzene	120-82-1	99%	2,000 µg/ml	+/- .04%
1,2,4-trimethylbenzene	95-63-6	99%	2,000 µg/ml	+/- .04%
1,3-dichlorobenzene	541-73-1	99%	2,000 µg/ml	+/- .04%
1,3-dichloropropane	142-28-9	99%	2,000 µg/ml	+/- .04%
1,3,5-trimethylbenzene	108-67-8	99%	2,000 µg/ml	+/- .04%
1,4-dichlorobenzene	106-46-7	99%	2,000 µg/ml	+/- .04%
2-chlorotoluene	95-49-8	99%	2,000 µg/ml	+/- .04%
2,2-dichloropropane	594-20-7	99%	2,000 µg/ml	+/- .04%
4-chlorotoluene	106-43-4	99%	2,000 µg/ml	+/- .04%
p-isopropyltoluene	99-87-6	99%	2,000 µg/ml	+/- .04%
benzene	71-43-2	99%	2,000 µg/ml	+/- .04%
bromobenzene	108-86-1	99%	2,000 µg/ml	+/- .04%
bromochloromethane	74-97-5	99%	2,000 µg/ml	+/- .04%

[1]Expiration date of the unopened ampul stored at recommended temperature.
[2]Listed in alphabetical order.
[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 Issued by DNV Certification, Inc.

Attachment 5 (continued)

TCL4 1 & 2 HIGH

RESTEK

CERTIFICATE OF ANALYSIS (cont.)

FOR LABORATORY USE ONLY–READ MSDS PRIOR TO USE.

PAGE 2

Cat. No.: 30431 Lot No.: A027506

Description: 502.2 CAL 2000 Mega-Mix

Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
bromodichloromethane	75-27-4	99%	2,000 µg/ml	+/- .04%
bromoform	75-25-2	99%	2,000 µg/ml	+/- .04%
carbon tetrachloride	56-23-5	99%	2,000 µg/ml	+/- .04%
chlorobenzene	108-90-7	99%	2,000 µg/ml	+/- .04%
chloroform	67-66-3	99%	2,000 µg/ml	+/- .04%
cis-1,2-dichloroethene	156-59-2	99%	2,000 µg/ml	+/- .04%
cis-1,3-dichloropropene	10061-01-5	99%	2,000 µg/ml	+/- .04%
dibromochloromethane	124-48-1	99%	2,000 µg/ml	+/- .04%
dibromomethane	74-95-3	99%	2,000 µg/ml	+/- .04%
ethylbenzene	100-41-4	99%	2,000 µg/ml	+/- .04%
hexachlorobutadiene	87-68-3	99%	2,000 µg/ml	+/- .04%
isopropylbenzene	98-82-8	99%	2,000 µg/ml	+/- .04%
methylene chloride	75-09-2	99%	2,000 µg/ml	+/- .04%
m-xylene	108-38-3	99%	2,000 µg/ml	+/- .04%
naphthalene	91-20-3	99%	2,000 µg/ml	+/- .04%
n-butylbenzene	104-51-8	99%	2,000 µg/ml	+/- .04%
n-propylbenzene	103-65-1	99%	2,000 µg/ml	+/- .04%
o-xylene	95-47-6	99%	2,000 µg/ml	+/- .04%
p-xylene	106-42-3	99%	2,000 µg/ml	+/- .04%
sec-butylbenzene	135-98-8	99%	2,000 µg/ml	+/- .04%
styrene	100-42-5	99%	2,000 µg/ml	+/- .04%
tetrachloroethene	127-18-4	99%	2,000 µg/ml	+/- .04%
tert-butylbenzene	98-06-6	99%	2,000 µg/ml	+/- .04%
toluene	108-88-3	99%	2,000 µg/ml	+/- .04%
trans-1,2-dichloroethene	156-60-5	99%	2,000 µg/ml	+/- .04%
trans-1,3-dichloropropene	10061-02-6	97%	2,000 µg/ml	+/- .04%
trichloroethene	79-01-6	99%	2,000 µg/ml	+/- .04%
Solvent: purge & trap methanol	67-56-1	99%		

[1]*Expiration date of the unopened ampul stored at recommended temperature.*

[2]*Listed in alphabetical order.*

[3]*Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.*

[4]*Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).*

Attachment 5 (continued)

TCL4 - GASES HIGH

RESTEK

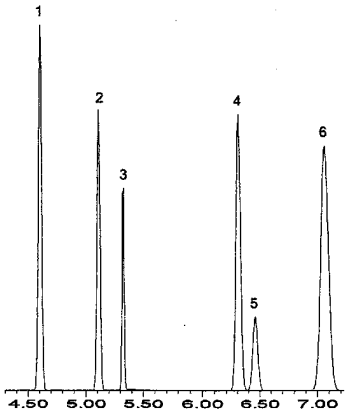
CERTIFICATE OF ANALYSIS

110 Benner Circle
Bellefonte, PA 16823-8812
Tel.: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY--READ MSDS PRIOR TO USE.

Cat. No.: 30042 Lot No.: A028050
Description: 502.2 Calibration Mix #1
Expiration Date[1]: March 2010 Storage: Freezer

Elution Order	Compound	CAS#	Percent Purity[3]	Concentration (weight/volume)[4]	Percent Uncertainty[5]
1	Dichlorodifluoromethane	75-71-8	99%	1,997 µg/ml	± 1.9%
2	Chloromethane	74-87-3	99%	2,003 µg/ml	± 1.3%
3	Vinyl chloride	75-01-4	99%	2,003 µg/ml	± 1.4%
4	Bromomethane	74-83-9	99%	1,997 µg/ml	± 1.3%
5	Chloroethane	75-00-3	99%	2,003 µg/ml	± 1.5%
6	Trichlorofluoromethane	75-69-4	99%	2,000 µg/ml	± 0.3%
Solvent:	Purge and trap Methanol	67-56-1	99%		



Column: 105m .32mm 1.8µm
Rtx-502.2 (cat.#10921)
Carrier gas: helium @ 2.2 ml/min.
Temp. program: 50°C (hold 12 min.)
Isothermal
Inj. temp.: 240°C
Det. temp.: 240°C
Detector type: MSD

John Lidgett
John Lidgett—QA Analyst

[1]Expiration date of the unopened ampul stored at recommended temperature.
[2]Listed in alphabetical order.
[3]Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
[4]Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
[5]Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED RESTEK QUALITY SEPARATES US FROM THE REST

MANUFACTURED UNDER RESTEK'S ISO 9001 REGISTERED QUALITY SYSTEM: Certificate #97-HOU-AQ-8550 issued by DNV Certification, Inc.

Attachment 5 (continued)

.TCL4 - KETONES

125 Market Street
New Haven, CT 06513
USA

AccuStandard, Inc.

Tel (203)786-5290
Fax (203)786-5287
Website AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: S-9863-R3
DESCRIPTION: Custom VOA Std #2
LOT: 209051016
SOLVENT: Methanol

EXPIRATION: Sep 6, 2009

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Acetone	67-64-1	100	1252	1252
Methyl ethyl ketone	78-93-3	99.9	1252	1251
4-methyl-2-pentanone	108-10-1	100	1252	1252
2-hexanone	591-78-6	99.4	1252	1244
Vinyl acetate	108-05-4	99.9	500.3	499.8
2-Chloroethylvinyl ether	110-75-8	99.5	500.0	497.5

6 Components

AccuStandard

1. All weights are traceable through NIST, Test No. 822/272103-05

2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±4% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).

3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

Certified by: R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005 and certified to ISO 9001:2000

OR-ORG/INO-001
Rev. 9/08

Attachment 5 (continued)

TCL4 – APPIX

125 Market Street
New Haven, CT 06513
USA

AccuStandard, Inc.

Tel (203)786-5290
Fax (203)786-5287
Website AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: S-9862-R2
DESCRIPTION: Custom VOA Std #1
LOT: B9020309
SOLVENT: Methanol

EXPIRATION: Jul 4, 2009

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (mg/mL)	Certified Analyte Concentration[2] (mg/mL)
Acetonitrile	75-05-8	99.9	0.504	.503
Allyl chloride	107-05-1	99.1	0.505	.500
1-Chlorohexane	544-10-5	99.0	0.505	.500
Chloroprene	126-99-8	97.0	0.520 *	.504
trans-1,4-Dichloro-2-butene	110-57-6	100	2.002	2.002
p-Dioxane	123-91-1	99.7	25.004	24.929
Ethyl methacrylate	97-63-2	99.5	5.002	[illegible]
Isobutyl alcohol	78-83-1	100	25.008	25.[illegible]
Methacrylonitrile	126-98-7	100	5.003	5.00[illegible]
Methyl iodide	74-88-4	99.5	0.504	.50[illegible]
Methyl methacrylate	80-62-6	99.8	5.002	[illegible]
Pentachloroethane	76-01-7	95.4	0.528 *	.504
Propionitrile	107-12-0	98.9	25.[illegible]	24.733
Acrolein	107-02-8	90.0	[illegible]	5.015
Acrylonitrile	107-13-1	99.8	[illegible]	4.991

15 Components

1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±4% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Certified by: R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005 and certified to ISO 9001:2000

OR-ORG/INO-001
Rev. 9/08

AccuStandard

Attachment 5 (continued)

TCL4 1&2 Additions
received 10 vials 12-19-08

RESTEK

Certificate of Composition

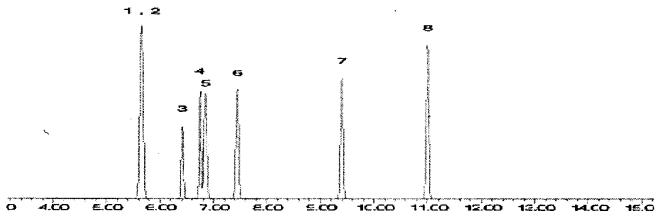
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 557093 **Lot No.:** A064665
Description : Custom 502.2 Additions Mix
Expiration Date[1]: December 2009 **Storage:** Freezer

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	1,1,2-Trichlorotrifluoroethane	76-13-1	97%	2,000.140 ug/ml	+/-0.08 %
2	1,1,1-Trichlorotrifluoroethane	354-58-5	98%	2,000.180 ug/ml	+/-0.08 %
3	Methyl acetate	79-20-9	99%	2,000.000 ug/ml	+/-0.08 %
4	Carbon disulfide	75-15-0	99%	2,000.000 ug/ml	+/-0.08 %
5	Methyl-tert-butyl ether (MTBE)	1634-04-4	99%	2,000.000 ug/ml	+/-0.08 %
6	Diisopropyl ether (DIPE)	108-20-3	99%	2,000.000 ug/ml	+/-0.08 %
7	Cyclohexane	110-82-7	99%	2,000.000 ug/ml	+/-0.08 %
8	Methylcyclohexane	108-87-2	99%	2,000.000 ug/ml	+/-0.08 %
Solvent:	P&T Methanol	67-56-1	99%		

Column: 105m x .32mm x 1.8um Rtx-502.2 (cat.#10921)
Carrier Gas: helium @ 2.2 ml/min.
Temp. Program: 40°C (hold 2 min.) to 240°C @ 8°C/min.
Inj. Temp: 200°C
Det. Temp: 250°C
Det. Type: MSD



Adam K Clark

Balance: 1125113331

Manufactured under Restek's ISO 9001:2000 Registered Quality System Certificate #FM80397

1 Expiration date of the unopened ampul stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preperation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

Attachment 6
DoD-QSM LCS Control Limits and Marginal Exceedances

CAS#	Volatile Compound	Soil CL	Soil ME
630-20-6	1,1,1,2-Tetrachloroethane	75-125	65-135
71-55-6	1,1,1,-Trichloroethane	70-135	55-145
79-34-5	1,1,2,2-Tetrachloroethane	55-130	40-145
79-00-5	1,1,2-Trichloroethane	60-125	50-140
75-34-3	1,1-Dichloroethane	75-125	65-135
75-35-4	1,1-Dichloroethene	65-135	55-150
563-58-6	1,1-Dichloropropene	70-135	60-145
87-61-6	1,2,3-Trichlorobenzene	60-135	50-145
96-18-4	1,2,3-Trichloropropane	65-130	50-140
120-82-1	1,2,4-Trichlorobenzene	65-130	55-140
95-63-6	1,2,4-Trimethylbenzene	65-135	55-145
96-12-8	1,2-Dibromo-3-Chloropropane	40-135	25-150
106-93-4	1,2-Dibromoethane	70-125	60-135
95-50-1	1,2-Dichlorobenzene	75-120	65-125
107-06-2	1,2-Dichloroethane	70-135	60-145
78-87-5	1,2-Dichloropropane	70-120	65-125
108-67-8	1,3,5-Trimethylbenzene	65-135	55-145
541-73-1	1,3-Dichlorobenzene	70-125	65-135
142-28-9	1,3-Dichloropropane	75-125	70-130
106-46-7	1,4-Dichlorobenzene	70-125	65-135
594-20-7	2,2-Dichloropropane	65-135	55-145
78-93-3	2-Butanone	30-160	10-180
95-49-8	2-Chlorotoluene	70-130	60-140
591-78-6	2-Hexanone	45-145	30-160
106-43-4	4-Chlorotoluene	75-125	65-135
108-10-1	4-Methyl-2-pentanone	45-145	30-165
67-64-1	Acetone	20-160	10-180
71-43-2	Benzene	75-125	65-135
108-86-1	Bromobenzene	65-120	55-130
74-97-5	Bromochloromethane	70-125	60-135
75-27-4	Bromodichloromethane	70-130	60-135
75-25-2	Bromoform	55-135	45-150
74-83-9	Bromomethane	30-160	10-180
75-15-0	Carbon Disulfide	45-160	30-180
56-23-5	Carbon Tetrachloride	65-135	55-145
108-90-7	Chlorobenzene	75-125	65-130
75-00-3	Chloroethane	40-155	20-175
67-66-3	Chloroform	70-125	65-135
74-87-3	Chloromethane	50-130	40-140
156-59-2	cis-1,2-Dichloroethane	65-125	55-135
10061-01-5	cis-1,3-Dichloropropene	70-125	65-135
124-48-1	Dibromochloromethane	65-130	55-140
74-95-3	Dibromomethane	75-130	65-135
75-71-8	Dichlorodifluoromethane	35-135	15-155

ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 6 (continued)
DoD-QSM LCS Control Limits and Marginal Exceedances

CAS#	Volatile Compound	Soil CL	Soil ME
100-41-4	Ethylbenzene	75-125	65-135
87-68-3	Hexachlorobutadiene	55-140	40-155
89-82-8	Isoproplybenzene	75-130	70-140
108-38-3	m, p-Xylene	80-125	70-135
75-09-2	Methylene Chloride	55/140	40-155
1634-04-4	Methyl-tert-butyl ether	40-125	25-140
91-20-3	Naphthalene	40-125	25-140
104-51-8	n-Butylbenzene	65-140	50-140
103-65-1	n-Propylbenzene	65-135	50-145
95-47-6	o-Xylene	75-125	70-135
99-87-6	p-Isopropyltoluene	75-135	65-140
135-98-8	sec-Butylbenzene	65-130	50-150
100-42-5	Styrene	75-125	65-135
98-06-6	Tert-Butylbenzene	65-130	55-145
127-18-4	Tetrachloroethene	65-140	55-140
108-88-3	Toluene	70-125	60-135
156-60-5	Trans-1, 2-Dichloroethene	65-135	55-145
10061-02-6	Trans-1,3-Dichloropropene	65-125	55-140
79-01-6	Trichloroethene	75-125	70-130
75-65-4	Trichlorofluoromethane	25-185	10-215
75-01-4	Vinyl Chloride	60-125	45-140

Attachment 7

5 vials - received 3-26-09 Red Source TCL4 1&2

125 Market Street
New Haven, CT 06513
USA

AccuStandard, Inc.

Tel (203)786-5290
Fax (203)786-5287
Website AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: M-502A-R-10X **EXPIRATION:** Oct 21, 2011
DESCRIPTION: Volatile Organic Compounds - Liquids
LOT: B8100189 **See reverse for additional certification information.**
SOLVENT: MeOH

This product is guaranteed accurate to ±0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Benzene	71-43-2	99.5	2000	1990
Bromobenzene	108-86-1	99.5	2001	1991
Bromochloromethane	74-97-5	99.1	2003	1985
Bromodichloromethane	75-27-4	98.7	2001	1975
Bromoform	75-25-2	99.2	2001	1985
n-Butylbenzene	104-51-8	99	2001	1981
sec-Butylbenzene	135-98-8	100	2001	2001
tert-Butylbenzene	98-06-6	99.6	2000	1992
Carbon tetrachloride	56-23-5	100	2001	2001
Chlorobenzene	108-90-7	100	2000	2000
Chloroform	67-66-3	99.8	2001	1997
2-Chlorotoluene	95-49-8	98.9	2001	1979
4-Chlorotoluene	106-43-4	98.5	2000	1970
Dibromochloromethane	124-48-1	99.1	2001	1983
1,2-Dibromo-3-chloropropane	96-12-8	100	2000	2000
1,2-Dibromoethane	106-93-4	99.9	2007	2005
Dibromomethane	74-95-3	98.6	2001	1973
1,2-Dichlorobenzene	95-50-1	98.2	2000	1964
1,3-Dichlorobenzene	541-73-1	99.8	2000	1996
1,4-Dichlorobenzene	106-46-7	99.9	2000	1998
1,1-Dichloroethane	75-34-3	99.6	2001	1993
1,2-Dichloroethane	107-06-2	100	2000	2000
1,1-Dichloroethene	75-35-4	99.0	2000	1980
cis-1,2-Dichloroethene	156-59-2	99.9	2000	1998
trans-1,2-Dichloroethene	156-60-5	96.0	2085 *	2002
1,2-Dichloropropane	78-87-5	100	2001	2001
1,3-Dichloropropane	142-28-9	99.9	2000	1998
2,2-Dichloropropane	594-20-7	100	2000	2000
1,1-Dichloropropene	563-58-6	99.1	2000	1982
cis-1,3-Dichloropropene	10061-01-5	97.7	2150 *	2101
trans-1,3-Dichloropropene	10061-02-6	97.7	1945 *	1900
Ethylbenzene	100-41-4	99.2	2000	1984
Hexachlorobutadiene	87-68-3	98.0	2001	1961
Isopropylbenzene	98-82-8	100	2000	2000
p-Isopropyltoluene	99-87-6	99.4	2001	1989
Dichloromethane	75-09-2	99.9	2003	2001

1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±4% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Page 1 of 2 Certified by: R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005 and certified to ISO 9001:2000

OR-ORG/INO-001
Rev. 9/08

AccuStandard

Attachment 7 (Continued)

125 Market Street
New Haven, CT 06513
USA

AccuStandard, Inc.

Tel (203)786-5290
Fax (203)786-5287
Website AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: M-502A-R-10X **EXPIRATION:** Oct 21, 2011
DESCRIPTION: Volatile Organic Compounds - Liquids
LOT: B8100189 **See reverse for additional certification information.**
SOLVENT: MeOH

This product is guaranteed accurate to ±0.5% of the Certified Analyte concentration through the Expiration Date on the Label.

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Naphthalene	91-20-3	99.8	2000	1996
n-Propylbenzene	103-65-1	100	2000	2000
Styrene	100-42-5	99.7	2001	1995
1,1,1,2-Tetrachloroethane	630-20-6	98.6	2000	1972
1,1,2,2-Tetrachloroethane	79-34-5	98.6	2001	1973
Tetrachloroethene	127-18-4	100	2002	2002
Toluene	108-88-3	99.9	2001	1999
1,2,3-Trichlorobenzene	87-61-6	100	2001	2001
1,2,4-Trichlorobenzene	120-82-1	99.3	2001	1987
1,1,1-Trichloroethane	71-55-6	100	2000	2000
1,1,2-Trichloroethane	79-00-5	98.3	2000	1966
Trichloroethene	79-01-6	100	2001	2001
1,2,3-Trichloropropane	96-18-4	98.5	2001	1971
1,2,4-Trimethylbenzene	95-63-6	98.2	2000	1964
1,3,5-Trimethylbenzene	108-67-8	98.8	2000	1976
o-Xylene	95-47-6	99.0	2000	1980
m-Xylene	108-38-3	99.9	2001	1999
p-Xylene	106-42-3	99.9	2000	1998

54 Components

AccuStandard

1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±4% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and is identical to the same lot# without the suffix.

* Weight compensated to 100% purity

Page 2 of 2 Certified by: R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005 and certified to ISO 9001:2000

OR-ORG/INO-001
Rev. 9/08

Attachment 7 (Continued)

~~15~~ 10 vials received 6-20-08 2nd Source Gas

125 Market Street
New Haven, CT 06513 USA

AccuStandard, Inc.

Tel (203)786-5290 Fax (203)786-5287
Web AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: M-502B-10X
DESCRIPTION: Volatile Organic Cmpds - Gases
LOT: B8020151
SOLVENT: MeOH

EXPIRATION: Feb 21, 2011

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
Bromomethane	74-83-9	99.0	2000	1980
Chloroethane	75-00-3	99.7	2000	1994
Chloromethane	74-87-3	99.5	2010	2000
Dichlorodifluoromethane	75-71-8	98	2008	1968
Trichlorofluoromethane	75-69-4	99.0	2001	1981
Vinyl chloride	75-01-4	99.5	2010	2000

6 Components

1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±4% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (-1A, -2B, etc.) on its lot# has had its expiration date extended and identical to the same lot# without the suffix.

Certified by R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005

OR-ORG/INO-001
Rev. 7/07

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Attachment 7 (Continued)

Received 2 ampules 2-6-09

Certificate of Analysis

NSI Solutions, Inc. * 7517 Precision Drive, Suite 101 * Raleigh, NC 27617 * 1-800-234-7837

Compound Name: Acrolein
Part Number: Q-6087-O
Lot Number: 020509-01

Expiration Date: 05/09
Solvent: P&T MeOH
(FLAMMABLE, IRRITANT)

COMPOUND NUMBER	ANALYTE	CHROMATOGRAPHIC Purity	GRAVIMETRIC CONCENTRATION (ug/mL)
W-02-06	Acrolein	99.2%	15017

PREPARATION: Reported concentration value has been corrected for purity. Concentration is calculated from wt/vol or vol/vol measurements using microbalances calibrated with NIST traceable weights to 0.0001 g and or ASTM Class A volumetric glassware or calibrated equivalent labware.

STORAGE & HANDLING: Store at ≤ 5° C. Allow to equilibrate to room temperature before use.

Kristi Bright

Kristi Bright, Organic Chemist

Attachment 7 (Continued)

Received 2 vials 5-10-09

RESTEK

Certificate of Composition

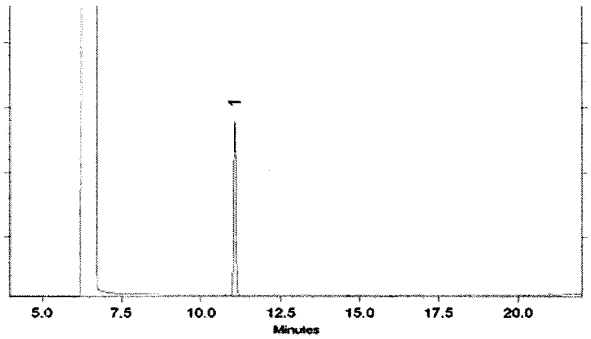
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 560347 **Lot No.:** A065489
Description : Custom Vinyl Acetate Standard
Expiration Date[1]**:** January 2011 **Storage:** Freezer

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	Vinyl acetate	108-05-4	98%	1,501.360 ug/ml	+/-0.12 %
Solvent:	P&T Methanol	67-56-1	99%		

Column: 105m x .53mm x 3.0um Rtx-502.2 (cat.#10910)
Carrier Gas: hydrogen @ 40 cm/sec.
Temp. Program: 40°C (hold 2 min.) to 240°C @ 8°C/min.
Inj. Temp: 200°C
Det. Temp: 250°C
Det. Type: FID



Diane Shaffer
Diane Shaffer - QA Analyst **Date Passed:** 01/27/2009 **Balance:** 1128353505 REVIEWED

Manufactured under Restek's ISO 9001-2000 Registered Quality System Certificate #FMB0397

1 Expiration date of the unopened ampule stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using one or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

Tech Tip:

Vinyl acetate is a volatile organic ester included in the target lists of several US EPA and other methods. Under acidic conditions, esters react with alcohols to form r esters (transesterification). Methanol-based mixes containing halogenated compounds are slightly acidic, so it is important to minimize exposure of vinyl acetate to n of halogenated compounds in methanol. For this reason, we offer vinyl acetate in individual solution, and suggest that it be introduced into the working level calibrati solution immediately before use. This will minimize problems and ensure more consistent results.

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Attachment 7 (Continued)

Received (2 vials) 3-10-09

RESTEK

Certificate of Composition

110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 560345 **Lot No.:** A065532
Description : Custom Ketones Plus Standard
Expiration Date[1]: July 2010 **Storage:** Freezer

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	Acetone	67-64-1	99%	3,750.000 ug/ml	+/-0.10 %
2	Acrylonitrile	107-13-1	99%	15,000.000 ug/ml	+/-0.10 %
3	2-Butanone (MEK)	78-93-3	99%	3,750.000 ug/ml	+/-0.10 %
4	2-Chloroethyl vinyl ether	110-75-8	98%	1,499.400 ug/ml	+/-0.10 %
5	4-Methyl-2-pentanone (MIBK)	108-10-1	99%	3,750.000 ug/ml	+/-0.10 %
6	2-Hexanone	591-78-6	99%	3,750.000 ug/ml	+/-0.10 %
Solvent:	P&T Methanol/Water (90:10)	67-56-1/7732-18-5	99%		

Column:
60m x .25mm x 1.4um
Rtx-502.2 (cat.#10916)

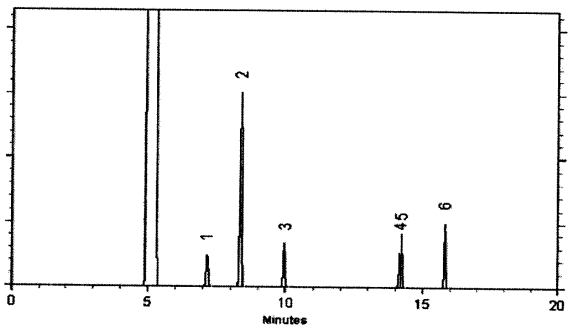
Carrier Gas:
helium @ 2.2 ml/min.

Temp. Program:
40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)

Inj. Temp:
200°C

Det. Temp:
250°C

Det. Type:
MSD



Sara Eyster
Sara Eyster QA Analyst

Date Passed: 01/29/2009 **Balance:** 1121472889

REVIEWED

Manufactured under Restek's ISO 9001-2000 Registered Quality System Certificate #FMB0397

1 Expiration date of the unopened ampule stored at recommended temperature.
2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage. In addition to detectors listed above, chemical identity and purity are confirmed using one or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details.
3 Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

Tech Tip:

Degradation of tetrachloroethylene to pentachloroethane may occur if solutions containing 2-chloroethyl vinyl ether are combined with solutions that contain tetrachloroethylene.

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Attachment 7 (Continued)

10 vials received 6-20-08 2nd Source TCL4 1#2. Additions

125 Market Street
New Haven, CT 06513 USA

AccuStandard, Inc.

Tel (203)786-5290 Fax (203)786-5287
Web AccuStandard.com

CERTIFICATE OF ANALYSIS

CATALOG NO: S-13035
DESCRIPTION: Custom Standard Mix
LOT: B8060071
SOLVENT: Methanol

EXPIRATION: Jun 9, 2010

See reverse for additional certification information.

This product is guaranteed accurate to ± 0.5% of the Certified Analyte concentration through the Expiration Date on the Label

Component	CAS #	Purity % (GC/FID)	Prepared Concentration[1] (µg/mL)	Certified Analyte Concentration[2] (µg/mL)
1,1,1-Trichlorotrifluoroethane	354-58-5	99	2005	1985
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	99.2	2001	1985
Carbon disulfide	75-15-0	99.9	2002	2000
MtBE	1634-04-4	99.9	2000	1998
Isopropyl ether	108-20-3	99.6	2005	1997
Cyclohexane	110-82-7	99.9	2001	1999
Methylcyclohexane	108-87-2	100	2001	2001
Methyl acetate	79-20-9	99.7	2014	2008

8 Components

1. All weights are traceable through NIST, Test No. 822/272103-05
2. Certified Analyte Concentration = Purity x Prepared Concentration. The Uncertainty calculated for this product is ±4% which is the Combined Uncertainty uc(y). It represents an estimated standard deviation equal to the positive square root of the total variance of the uncertainty of components. The Expanded Uncertainty is U which is Uc(y) *K where K is the coverage factor at the 95% confidence level (K=2).
3. A product with a suffix (1A, 2B, etc.) on its lot# has had its expiration date extended and identical to the same lot# without the suffix.

Certified by: R. Cooper

AccuStandard is accredited to ISO/IEC 17025:2005

OR-ORG/NO-001
Rev. 7/07

AccuStandard

Attachment 7 (Continued)

2nd Source
A [illegible]

Received 6-20-08

RESTEK

Certificate of Composition

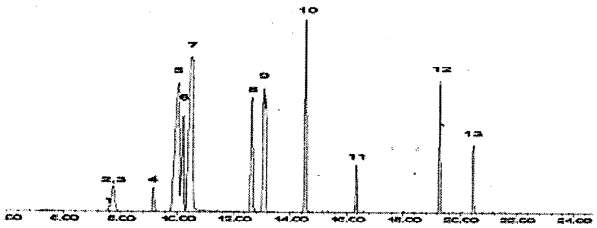
110 Benner Circle
Bellefonte, PA 16823-8812
Tel: (800)356-1688
Fax: (814)353-1309

FOR LABORATORY USE ONLY-READ MSDS PRIOR TO USE.

Catalog No. : 563125 **Lot No.:** A060833
Description : Custom VOA Standard #1
Expiration Date[1]**:** June 2009 **Storage:** Freezer

Elution Order	Compound	CAS #	Percent Purity [2]	Concentration [3] (weight/volume)	Percent Uncertainty [4]
1	Acetonitrile	75-05-8	99%	500.000 ug/ml	+/-0.05 %
2	Allyl chloride (3-chloropropene)	107-05-1	98%	499.800 ug/ml	+/-0.05 %
3	Iodomethane (methyl iodide)	74-88-4	99%	500.000 ug/ml	+/-0.05 %
4	Chloroprene (2-chloro-1,3-butadiene)	126-99-8	99%	485.770 ug/ml	+/-5.04 %
5	Methacrylonitrile	126-98-7	99%	5,000.000 ug/ml	+/-0.05 %
6	Propionitrile	107-12-0	99%	25,000.000 ug/ml	+/-0.05 %
7	Isobutanol (2-Methyl-1-propanol)	78-83-1	99%	25,000.000 ug/ml	+/-0.05 %
8	Methyl methacrylate	80-62-6	99%	5,000.000 ug/ml	+/-0.05 %
9	1,4-Dioxane	123-91-1	----%	25,000.000 ug/ml	+/-0.05 %
10	Ethyl methacrylate	97-63-2	99%	5,000.000 ug/ml	+/-0.05 %
11	1-Chlorohexane	544-10-5	99%	500.000 ug/ml	+/-0.05 %
12	trans-1,4-dichloro-2-butene	110-57-6	98%	2,000.180 ug/ml	+/-0.05 %
13	Pentachloroethane	76-01-7	98%	499.800 ug/ml	+/-0.05 %
Solvent:	P&T Methanol	67-56-1	99%		

Column: 60m x .25mm x 1.4um
Rtx-502.2 (cat.#10916)
Carrier Gas: helium @ 2 ml/min.
Temp. Program: 40°C (hold 2 min.) to 240°C
@ 8°C/min. (hold 10 min.)
Inj. Temp: 200°C
Det. Temp: 250°C
Det. Type: MSD



Sara Eyster
Sara Eyster / QA Analyst

Balance 1121472889

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3 Based upon gravimetric preparation with balance calibration verified using NIST traceable weights (seven mass levels).
4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

ISO 9001 REGISTERED

Manufactured under Restek's ISO 9001 Registered Quality System
Certificate #FM80397

Attachment 8

Condition Codes Used for Documentation in Logbooks

Condition Code	Definition
CS	Contamination Suspected
CT	Contamination Verified
DA	Dilution Acceptable
DI	Requires Dilution
DW	Dilution Wrong
ES	Extraction Same
EF2JS	Ending Standard Failed Second Tune Injection Same
JS	Injection Same
IH	Internals High
IL	Internals Low
IM	Internals Missing
LA	Lab Accident (Must Explain)
OT	Other (Must Explain)
OQ	Original Run Qualified
OW	Original Wrong for QC Sample
RP	Reportable Prior Run
RT	Retention Time Shift
RU	Run Unnecessarily
SF	Spike Recoveries Failed
SH	Surrogate Recoveries Failed High
SI	Spiked Inadvertently
SL	Surrogate Recoveries Failed Low
SM	Surrogate or Spike Missing
IF	Instrument Failure (Must Explain)
US	Unacceptable Standard (Must Explain)
VR	Verify Results (Must Explain)
NM	Did Not Match Prior Run or Duplicate
RN	Reanalyze Neat
PC	Poor Chromatography
WS	Wrong Standard

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Attachment 9

CompuChem

a division of Liberty Analytical Corporation

Notification Regarding Manual Editing/Integration Flags

In some instances, manual adjustments to the software output are necessary to provide accurate data. These manual integrations are performed by the data reviewers, GC/MS operators, or GC/HPLC chemists. An Extracted Ion Current Profile (EICP) or a GC/HPLC chromatographic peak has been provided for the manual integration performed on each compound to demonstrate the accuracy of that process. The manual integrations are flagged on the quantitation report in the far right column beyond the FINAL concentration for GC/MS analysis, and in the "Flags" column for GC/HPLC analysis. The manual editing/integration flags are:

M **- Denotes that a manual integration has been performed for this compound. The manual integration was performed in order to provide the most accurate area count possible for the peak. The most common reasons for performing manual integrations/editing are: the compound was not found by the automatic integration routine, the compound was incorrectly integrated by the automatic integration routine, and the co-eluting compounds were incorrectly integrated by the automatic integration routine.**

H **- Denotes that the data reviewer, GC/MS operator, or GC/HPLC Chemist has chosen an alternate peak within the retention time window from that chosen by the software for that compound. No manual integration is performed in choosing an alternate peak. The software still performs the integration.**

MH **- Denotes that an alternate peak has been chosen within the retention time window from that chosen by the software for that compound and also a manual integration of the chosen peak has been performed. The manual integration was performed in order to provide the most accurate area count possible for the peak.**

L **- Denotes that a data reviewer or GC/MS operator has selected an alternate library search. This is typically done when an additional tentatively identified compound (TIC) has been added to the number of peaks searched. No manual integration is performed in choosing an alternate peak. The software still performs the integration.**

ML **- Denotes that an alternate GC/MS library search has been selected and a manual integration has also been performed. This is typically done when an additional TIC has been added and the TIC peak also required a manual integration.**

These codes will appear in the GC/MS and GC/HPLC raw data.

Revision 8 (01/29/2011)

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SOP 2.2.3.1

Environmental Testing Laboratories
CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-1003 SOP Section #: 2.2.3.1 Revision #: 12

SOP Title: Preparation of Water Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3510C and TN DEC

Effective date: (QA fills in) 6/29/11

- Procedure prepared by: J-Rdp Date: 04/28/2011
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Manera Date: 05-04-2011
- Reason for change: Addition of LIMS Information; Change in Spiking information; Addition of DoD-QSM protocols
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Edition, Update III 12/96 Method 3510C; "Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID TN DEC, March 1999; DoD-QSM Version 4.2 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -1003: Preparation of Water Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3510C and TN DEC

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Sample Preparation Procedure -1003: Preparation of Water Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3510C and TN DEC

1.0 Scope and Application

This Standard Operating Procedure (SOP) is applicable to the preparation of aqueous samples by SW-846 Method 3510C for the analysis of extractable diesel range organics and jet fuels, by the GC technique using the procedures taken from SW-846 and TN DEC methodologies. DROs correspond to the range of alkanes from C_{10} to C_{28} (C_{12} to C_{40} for TN DRO) and covering a boiling point range of approximately 170 ^{o}C to 430 ^{o}C.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A measured volume of sample, approximately 1 liter, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and concentrated to a final volume of 2.5 mL (1.0 mL for TN DRO). The extract is then analyzed by Gas Chromatograph/Flame Ionization Detector (GC/FID). This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration

result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.4 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.5 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.6 Reporting Limit (RL) –

3.6.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.6.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.**7** A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single extraction batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 Extraction Batch – a group of to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.9 SC DHEC – South Carolina Department of Health and Environmental Control

3.10 **DoD**-QSM – Department of Defense Quality Systems Manual

3.11 Ohio VAP – Ohio Environmental Protection Agency Voluntary Action Program

3.12 Diesel fuel – consists primarily of straight-chain hydrocarbons (alkenes and alkanes) ranging in length from C10 to C28 (C12 to C40 for TN DRO). Carbon chain length of C16 and C17 predominate in the mixture, whose composition approximates a bell-shaped curve with C16 and C17 as the mean. Diesel fuel may also contain some aromatic constituents (depending on the source and refining process), including benzene. They usually account for less than 0.1% of the total product.

3.13 Marker Vial – a vial of same manufacture and size as sample extract vial containing the volume of liquid desired as a final volume for the sample extract. Marker vials are created using class 'A' pipettes.

3.14 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines which may cause misinterpretation of chromatographic

data. All of those materials must be demonstrated to be free from interferences under the conditions of analysis by running laboratory method blanks.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the site being sampled.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, i.e., extraction, safety glasses, gloves and lab coats are a minimum requirement. Sample extracts must be prepared under a hood. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and MSDS are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 Disposable pipets – 1 mL

6.2 Separatory Funnel – 2000 mL, with Teflon® stopcock

6.3 Kuderna-Danish (K-D) apparatus

6.3.1 Concentrator tube – 10 mL, graduated

6.3.2 Evaporative flask – 500 mL

6.3.3 Snyder column – three-ball macro

6.3.4 Snyder column – two ball micro

6.4 Silicon carbide boiling chips – approximately 10/40 mesh

6.4.1 Heat to 400 °C for 30 minutes or Soxhlet extract with methylene chloride prior to use.

6.5 Water bath – heated, with concentric ring cover, capable of temperature control of ± 2 °C. The bath must be located under a ventilation hood.

6.6 Vials – with Teflon®-lined septa and screw caps or crimp-tops, 5 mL amber and **2** mL amber

6.7 Nitrogen evaporation device – equipped with a water bath that can be maintained at 35-40 °C. The N-Evap by Organomation Associates, Inc. (South Berlin, MA) or equivalent is suitable.

6.8 Pyrex glass wool – furnaced

6.9 Erlenmeyer flask – 250 mL

6.10 Graduated cylinder, clear glass – 1 L or 2 L, Class B, 3-5% tolerance

6.11 Graduated glass jar – 1 L

6.12 Element DataSystem® LIMS computer software by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook (22F) or Promium Element® LIMS.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2-6 ^{o}C in the reach-in cold storage units in the preparation and organic standards laboratories when not in use. **Spiking solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.**

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: All standards and reagents are subject to change in vendor and in concentration. **Standards may be purchased as certified neat materials or concentrated solutions. All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.** The Reporting Limits

are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Sodium sulfate, Mallinckrodt anhydrous, reagent grade, or equivalent

7.2.1 Heat sodium sulfate at 400 °C for four hours**. Cool**, and store in a glass bottle.

7.3 Methylene chloride, pesticide grade or equivalent.

7.4 Methanol – pesticide grade or equivalent

7.5 Stock Standards

7.5.1 Stock Diesel Spiking Solution – NSI Diesel Fuel #2 (Catalog # UST-145) – 50,000 µg/mL

7.5.2 Stock Restek Motor Oil Standard (Catalog# 31464) – 50,000 µg/mL

7.5.3 DRO Surrogate Stock Solution – NSI o-Terphenyl (Catalog # W-1341H) – 10,000 µg/mL.

7.5.4 Stock solution expiration dates are provided by the manufacturer

7.6 Working Standards

7.6.1 Diesel Spiking Solution # 4028

7.6.1.1 Prepare the Diesel Spiking Solution # 4028 by adding 2.5 mL of NSI Diesel Fuel #2 to methanol in a 25 mL volumetric flask. Dilute to volume with methanol. The concentration of the resulting solution is 5000 µg/mL. **Prepare this solution fresh every six months or sooner if degradation occurs.**

7.6.2 **Motor** Oil Spiking Solution **(ORO)**

7.6.2.1 Prepare the **Motor** Oil Spiking Solution by adding 1.0 mL of Stock Restek Motor Oil to acetone in a 10 mL volumetric flask. Dilute to volume with acetone. The concentration of the resulting solution is 5000 µg/mL. **Prepare this solution fresh every six months or sooner if degradation occurs.**

7.6.**3** DRO Surrogate Solution #444

7.6.**3**.1 Prepare the DRO Surrogate Solution #444 by adding 5.0 mL of NSI o-Terphenyl to methanol in a 100 mL volumetric flask. Dilute to volume with methanol. The concentration of the resulting solution is 500 µg/mL. **Prepare this solution fresh every six months or sooner if degradation occurs.**

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples."

8.2 All samples prepared by this SOP must be extracted within 7 days of collection.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared with each extraction batch.

9.2 Laboratory Control Sample (LCS)

9.2.1 A laboratory control sample must be prepared with each extraction batch.

9.2.2 Duplicate LCSs must be prepared with each extraction batch for TN DRO.

9.3 Matrix Spike, Matrix Spike Duplicate, and Duplicate Sample

9.3.1 A matrix spike (MS) and matrix spike duplicate (MSD) are prepared for each SDG batch.

9.3.1.1 **DoD**-QSM and SC DHEC require that a matrix spike (MS) and matrix spike duplicate (MSD) be prepared with each extraction batch.

9.3.2 A duplicate sample may be prepared instead of the MSD, if samples are expected to contain target compounds.

10.0 Calibration and Standardization

N/A

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The sample preparation technician must complete the extraction **preparation bench** sheet (**See example** Attachment 1). Any unused portion of the **bench** sheet must be **"**Z'd**"** out. The laboratory supervisor or his/her designee reviews the completed **bench** sheet for completeness and accuracy, and then signs and **dates** the **bench** sheet. The **bench sheet** accompanies the samples to the analytical laboratory. Include on the **bench** sheet the manufacturer and lot number of reagents/solvents used. If samples are to be analyzed for ORO only substitute ORO spiking solution for DRO spiking solution in the following instructions. If samples are to be analyzed for DRO and ORO add ORO spiking solution in addition to DRO spiking solution in the following instructions.

11.1 Rinse all glassware to be used in this procedure with methylene chloride prior to use. Dispose of the rinse solvent into the appropriately labeled waste container.

11.2 Allow samples to equilibrate to room temperature before taking aliquots.

11.3 Sample Preparation

11.3.1 Thoroughly mix the field sample by shaking the sample container before removing an aliquot.

11.3.2 Prepare the sample for extraction by measuring a 1 L aliquot using a graduated cylinder or graduated jar. Carefully pour the sample into a 2 L separatory funnel. Add 5 to 10 mL of methylene chloride to the graduated cylinder or graduated jar to rinse. Add the rinse to the separatory funnel. Add 0.5 mL of DRO Surrogate Solution #444 to the sample in the funnel.

11.3.2.1 If the entire contents of the sample container are used, Rinse the sample container with 5 to 10 mL of methylene chloride. Add the rinse to the separatory funnel.

11.3.3 Prepare the method blank by measuring 1 L of reagent water into a separatory funnel. Add 0.5 mL of DRO Surrogate Solution #444 to the blank in the funnel.

11.3.4 Prepare the MS and MSD by measuring two 1 L aliquots of the designated sample into two separatory funnels. Add 0.5 mL of DRO Surrogate Solution #444 and **0.5 mL** of Diesel Spiking Solution #4028 to each sample.

11.3.5 Prepare a LCS by measuring 1 L of reagent water into a separatory funnel. Add **0.5 mL** of Diesel Spiking Solution #4028 and 0.5 mL of Surrogate Solution #444.

11.4 Sample Extraction

11.4.1 Add 60 mL of methylene chloride to each separatory funnel. Extract all samples by shaking the separatory funnels for 2 minutes, periodically venting the funnel to release excess pressure.

11.4.2 Allow the solvent layer to separate from the water layer for a minimum of 10 minutes. If emulsions are present, centrifuge techniques or other appropriate manual techniques must be employed to achieve adequate separation.

11.4.2.1 To break an emulsion using centrifugation, drain the emulsion into a centrifuge bottle. Place the bottle in the centrifuge. (Always use a bottle with approximately the volume of methylene chloride as a counter-weight, if only one sample is being centrifuged). Secure the lid of the centrifuge and start. Let the sample(s) spin for 2 minutes at 2000 RPM and turn off the centrifuge. Once the sample(s) have stopped spinning, remove the bottles. Pour the sample extract back into the appropriate separatory funnel. Rinse the bottle with 5 to 10 mL of methylene chloride and pour the rinse into the separatory funnel.

11.4.3 Drain the methylene chloride layer through sodium sulfate in a powder funnel into a 250 mL Erlenmeyer flask.

11.4.4 Repeat **Sections** 11.4.1 through 11.4.3 two more times**,** combining all three portions of methylene chloride extract in the Erlenmeyer flask.

11.4.5 Assemble a K-D concentrator by attaching a 10 mL concentrator tube to a 500 mL evaporative flask.

11.4.6 Pour the combined extracts into the K-D concentrator. Rinse the Erlenmeyer flask with 10 to 20 mL of methylene chloride to complete the transfer.

11.5 Extract Concentration

11.5.1 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Wet the Snyder column by adding approximately 1 mL of methylene chloride to the top.

11.5.2 Place the K-D on a hot water bath so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. The temperature of the water bath should be **between 80-90** °C or adjusted as required to complete the concentration in 15 to 20 minutes.

11.5.3 When the apparent volume of the extract reaches **2** mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.6 Final Extract Concentration

11.6.1 Micro Snyder Column Technique

11.6.1.1 Attach a 2-ball micro Snyder column to the concentrator tube. Wet the Snyder column by adding about 1-mL of methylene chloride to the top. Concentrate the extract by holding the concentrator tube over the hot water bath. When the extract reaches an apparent volume of **0.5** mL, remove it from the bath. Allow the apparatus to cool and drain.

11.6.2 Nitrogen Evaporation Technique

11.6.2.1 Remove the Snyder column and place the concentrator tube on the nitrogen evaporation apparatus (Organomation) and concentrate to an apparent volume of 0.5 mL (0.3 mL for TN DRO).

11.7 Qualitatively transfer the extract to a 5 mL screw-top vial. Pipet the extract into the vial. Rinse the concentrator tube with approximately 1 mL of methylene

chloride and add the rinse to the vial. Adjust the extract to a final volume of 2.5 mL with methylene chloride using the 2.5 mL Marker Vial.

11.7.1 For TN DRO extracts, pipet**te** the extract into a **2** mL vial. Rinse the concentrator tube with approximately 0.5 mL of methylene chloride and add the rinse to the vial. Adjust the final extract volume to 1.0 mL using the 1.0 mL Marker Vial.

11.8 Attach a label to the vial. The label must contain the following information:

CompuChem Number:	XXXXXX
Method:	**-8015**
Date Extracted:	XX/XX/XX

11.9 Complete the extraction **preparation bench** sheet for the **Method** (**See example** Attachment 1). Include on the **bench** sheet the manufacturer and lot number of reagents/solvents used. Any unused portion of the **bench sheet** must be **"**Z'd**"** out. The laboratory supervisor or his/her designee must review the **bench sheet** for completeness and accuracy, and then sign and date the **bench** sheet.

11.10 Deliver the batch of labeled extracts, with the completed extraction **preparation bench** sheet and accompanying chain-of-custody form, to the GC analytical laboratory and place them in the designated cold storage unit.

12.0 Data Analysis and Calculations

N/A

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained in electronic form on intranet QA server and in hardcopy form in the QA department or in the data storage room.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible,

laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3510C

16.2 "Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID", TN DEC, March 1999

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 NELAC Standards, effective July 2003, plus revisions

16.5 EPA QA/G6: Guidance for Preparing of Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007

16.6 CompuChem Quality Manual, Revision **13**, **November 17**, **2009** plus revisions

16.7 Sample Control SOP 4.6, "Storing Samples"

16.8 Determinative Chromatographic Separations, SW-846 3rd Edition, Update IV, March, 2003, Method 8000C

16.9 Non-Halogenated Organics by Gas Chromatography, SW-846 3rd Edition, Update IV, Method 8015C, February 2007

16.10 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010, plus revisions

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example Extraction Preparation Bench Sheet**

Attachment 1

Assigned To _____

Employee ID Number _____

Matrix: Water

PREPARATION BENCH SHEET

1022802

GC-8015C DRO DOD

Prepared using: GC - EPA 3510C_GC

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial (mL)	Final (uL)	QC	Surr (uL)	Comments
1022802-BLK1	PBLKWI	Blank				500	
1022802-BS1	PWILCS	LCS				500	
1022802-MS1	1738MW01-10DMS	Matrix Spike				500	
1022802-MSD1	1738MW01-10DMSD	Matrix Spike Dup				500	
1102094-01	1738MW01-10D	Sample			QC	500	
1102094-02	2842BMW01-10D	Sample				500	
1102094-03	2842BMW06-10D	Sample				500	
1102094-04	1738MW02-10D	Sample				500	
1102094-05	1738MW03-10D	Sample				500	
1102094-06	1738MW05R-10D	Sample				500	
1102094-07	1738MW01D-10D	Sample				500	
1102094-08	735MW05-10D	Sample				500	
1102094-09	735MW06-10D	Sample				500	
1102094-10	AOCF-FB01-10D	Sample				500	
1102094-11	AOCF-ER01-10D	Sample				500	
1102094-12	AOCF-ER02-10D	Sample				500	
1102094-13	AOCF-ER03-10D	Sample				500	
1102094-15	AOCF-ER04-10D	Sample				500	

	Description	Spike Amount (uL)	Lot Number
SURROGATE	444	500	
SPIKE	4028 DRO SPK	500	LCS/LCSD
SPIKE	4028 DRO SPK	500	MS/MSD

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

Manuf. and lot # of reagents/solvents used: _____

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Note: This page subject to change without notice.

SOP 2.2.3.7

CompuChem
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Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: 1PP SOP Section #: 2.2.3.7 Revision #: 13

SOP Title: Analysis of Diesel Range Organics (DRO), Oil Range Organics (ORO) JP4, JP8, and Total Jet Fuel in Aqueous and Solid Samples by SW-846 and Extractable Petroleum Hydrocarbons by TN DEC

Effective date: (QA fills in) 10-25-11

- Procedure prepared by: Teresa Cimmett Jennings Date: 10-24-11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 10-24-11
- Reason for change: Annual Review
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Ed., Update III, Method 8015B; SW-846, 3RD Ed., Update IV, Method 8015C; Method for Determination of Extractable Petroleum Hydrocarbons GC/FID, TN DEC, March, 1999; DoD QSM Version 4.2, 10/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure — Analysis of Diesel Range Organics (DRO), Oil Range Organics (ORO), JP4, JP8, and Total Jet Fuel in Aqueous and Solid Samples by SW-846 and Extractable Petroleum Hydrocarbons by TN DEC

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If words above are not highlighted, this is an uncontrolled copy of this document.

Instrument Procedure — Analysis of Diesel Range Organics (DRO) Oil Range Organics (ORO), JP4, JP8, and Total Jet Fuel in Aqueous and Solid Samples by SW-846 and Extractable Petroleum Hydrocarbons by TN DEC

1.0 Scope and Application

This method is used for the determination of total petroleum hydrocarbons (TPH) including diesel range organics corresponding to the range of alkanes from C_{10} to C_{28} (C_{12} to C_{40} for TN DRO) and covering a boiling point range of 170 ^{o}C to 430 ^{o}C. ORO, JP4, JP8, and Total Jet Fuel are determined by the retention time window of the chromatographic "fingerprint" pattern of the fuel standard to be determined. Constituents are extracted from aqueous and solid samples into a solvent amenable to analysis by a gas chromatography (GC) utilizing a flame ionization detector (FID).

The reporting limit is at or above the lowest calibration standard concentration. The reporting limits are as follows:

- DRO EPA 8015C – 0.5 mg/L and 10 mg/Kg
- TN DRO (by TN DEC)– 0.2 mg/L and 8.0 mg/Kg
- ORO – 1.0 mg/L and 10 mg/Kg
- JP4 and JP8 – 0.2 mg/L and 10 mg/Kg

Staff members performing the procedures described in this Standard Operating Procedure (SOP) are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies. Refer to the Quality Manual for procedures for performing and documenting demonstration of capability.

2.0 Summary of Method

2.1 Diesel **Range Organic** samples are prepared by **SOP 2.2.3.1, "Preparation of Water Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3510C and TN DEC" or SOP 2.2.3.8, "Preparation of Soil/Sediment/Sludge Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3550B, Method 3550C, and DRO by TN DEC."** and then introduced into the GC and analyzed. An appropriate column and temperature program is used in the gas

chromatograph to separate the organic compounds. Detection is achieved by a flame ionization detector (FID). Fused silica capillary columns are necessary for the analysis of petroleum hydrocarbons. Quantitation is by means of the external standard technique. Quality is measured through surrogate recovery, LCS**,** and matrix spike precision and accuracy.

2.2 This procedure encompasses EPA 8015C. This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

3.0 Definitions

3.1 Method Detection Limit (MDL) – The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) – The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1%. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of the measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL)

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported as qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Diesel Range Organics (DRO) – consists primarily of straight-chain hydrocarbons (alkenes and alkanes) ranging in length from C_{10} to C_{28} (C_{12} to C_{40} for TN DRO). Carbon chain length of C_{16} and C_{17} predominate in the mixture, whose composition approximates a bell-shaped curve with C_{16} and C_{17} as the mean (see Attachment 3). Diesel Range Organics may also contain some aromatic constituents (depending on the source and refining process), including benzene. They usually account for less than 0.1% of the total product.

3.7 Reporting Units – mg/L for aqueous samples; mg/Kg for solid samples

3.8 A sample delivery group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

Note: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.9 Extraction Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.10 SC DHEC – South Carolina Department of Health and Environmental Control

3.11 **DoD**-QSM – Department of Defense Quality Systems Manual

3.12 TN-DEC – Tennessee Department of Environment and Conservation

3.13 Ohio VAP – Ohio Environmental Protection Agency Voluntary Action Program

3.14 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Contamination by carryover can occur when high-concentration and low-concentration samples are analyzed in sequence. The sample syringe must be rinsed out with methylene chloride between samples.

4.2 Solvents, reagents, glassware, and other sample processing hardware must be demonstrated to be free from interferences under the conditions of the analysis by running method blanks and solvent blanks.

4.3 Due to the nature of the solid and water matrices produced by these samples, the most common problem encountered analytically is interference from other petroleum by-products encountered with the diesel **range organics**. Maintenance procedures are employed when poor column resolution results from dirty injector liners or dirty injector ports. Instrument maintenance logbooks are maintained for each GC instrument.

5.0 Safety

5.1 The extracted samples must be handled with care due to the toxicity **of methylene** chloride and the possible high levels of TPH extractable contamination. Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves**,** and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 GC System

6.1.1 Varian 3400 **and HP 5890** gas chromatographs **are** suitable for on-column and splitless injection with all required accessories, including syringes, analytical columns, gases, FID detectors and data system.

6.2 Column

6.2.1 GC column – Restek Rtx-5MS: 30 m, 0.53 mm I.D. (or equivalent).

6.3 Supplies

6.3.1 Pipets – Class A glass pipets, assorted sizes.

6.3.2 Pasteur pipets

6.3.3 Gas-tight syringes – assorted sizes.

6.3.4 Volumetric flasks – Class A glass in assorted sizes.

6.3.5 Vials or Bottles – assorted sizes with Teflon-lined caps

6.3.6 Drummond (or equivalent) adjustable pipet with disposable capillary tubes to deliver volumes of 20 - 100 µL

6.4 Data system: Standards and samples are processed using the EZ Chrom® Elite data acquisition system, Target® Quick Forms report generation software from ThruPut Systems, and Element DataSystem® LIMS by Promium, plus revisions

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook (22F) or Promium Element LIMS.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2 - 6 ^{o}C in the laboratory when not in use. Stock solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.

Note: All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration. All reagents and standards must be ACS (American Chemical Society) grade or equivalent**, unless otherwise denoted**.

7.1 Reagent water – All water used in this procedure must be reagent-grade Type I water with regard to resistivity of > 10 mega ohm-cm (19th and 20th Editions of Standard Methods, Method 1080) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP).

7.2 Methylene Chloride – Reagent Grade

7.3 Acetone – Reagent Grade

7.4 Methanol – Reagent Grade

7.5 Stock Standards Standard Solutions

7.5.1 NSI o-terphenyl standard (Catalog# 1341H) – 10,000 µg/mL of o-terphenyl in methylene chloride.

7.5.2 Restek o-terphenyl standard (Catalog# 31097) - 10,000 µg/mL of o-terphenyl in methylene chloride).

7.5.3 Restek Diesel Fuel #2 Standard (Catalog# 31258) – 50,000 µg/mL diesel fuel #2 in methylene chloride.

7.5.4 NSI Diesel Fuel #2 Standard (Catalog# UST-145) - 50,000 µg/mL diesel fuel #2 in methylene chloride.

7.5.5 Restek Motor Oil Standard (Catalog# 31464) – 50,000 µg/mL motor oil in methylene chloride.

7.5.6 Restek JP-4 Military Fuel (Catalog# 31250) – 50,000 µg/mL JP-4 in methylene chloride.

7.5.7 Restek JP-8 Military Fuel (Catalog# 31254) – 50,000 µg/mL JP-8 in methylene chloride.

7.5.8 NSI Florida TPH Mix (Catalog# C-443) – 2000 µg/mL each compound (C_8, C_{10}, C_{12}, C_{14}, C_{16}, C_{18}, C_{20}, C_{22}, C_{24}, C_{26}, C_{28}, C_{30}, C_{32}, C_{34}, C_{36}, C_{38}, and C_{40}) in methylene chloride). This standard is used as the DRO/**ORO** retention time (RT) Marker.

7.5.9 Restek Diesel Range Organics Mix (EPA/WISC) (Catalog# 31064) – 2000 µg/mL each compound (C_{10}, C_{12}, C_{14}, C_{16}, C_{18}, C_{20}, C_{22}, C_{24}, C_{26}, C_{28}) in methylene chloride). This standard is used as the DRO RT Marker.

7.6 Intermediate Calibration Standard Solutions

7.6.1 DRO Calibration Standard (**CAL5**)

7.6.1.1 Prepare a 5,000 µg/mL DRO standard solution by adding 2.5 mL of **Restek** Diesel Fuel #2 Mix and 1.25 mL NSI of o-terphenyl surrogate stock to a 25 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.6.2 DRO/ORO (**CAL5**) (includes TN DRO)

7.6.2.1 Prepare a 5,000 µg/mL DRO/ORO standard solution by adding 2.5 mL of Restek Motor Oil Standard, 2.5 mL of **Restek** Diesel Fuel #2 Mix, and 1.25 mL of NSI o-terphenyl surrogate stock to a 25 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.6.3 JP-4 Calibration Standard (**CAL5**)

7.6.3.1 Prepare a 5,000 µg/mL JP-4 standard solution by adding 2.5 mL of Restek JP-4 and 1.25 mL of NSI o-terphenyl surrogate stock to a 25 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.6.4 JP-8 Calibration Standard (**CAL5**)

7.6.4.1 Prepare a 5,000 µg/mL JP-8 standard solution by adding 2.5 mL of Restek JP-8 and 1.25 mL of NSI o-terphenyl surrogate stock to a 25 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.6.5 Retention Time Marker Standard (RTMARK)

7.6.5.1 For TN DRO: Prepare a 50 µg/mL Retention Time Marker standard solution by adding 1.25 mL of NSI Florida TPH Mix

(see Section 7.5.8) to a 50 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.6.5.2 For DRO: Prepare a 50 µg/mL Retention Time Marker standard solution by adding 1.25 mL of Restek DRO Mix (EPA/WISC) (see Section 7.5.9) to a 50 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.6.6 Instrument Blank (IBLK)

7.6.6.1 Prepare a 100 µg/mL o-terphenyl solution by adding 1.0 mL of NSI surrogate stock solution to a 100 mL volumetric flask containing about 10 mL of methylene chloride. Dilute to volume with methylene chloride. Prepare this standard every 6 months.

7.7 Working Calibration Standard Solutions

7.7.1 Five-Point Calibration Standards

A five-point calibration curve is prepared by making 2, 5, 10, and 25 times dilutions of the high calibration standard in methylene chloride resulting in the standard concentrations outlined in the following table.

Standard	**CAL 5 (neat) µg/mL**	**CAL 4** 250 **(2x) µg/mL**	**CAL 3 (5x) µg/mL**	**CAL 2 (10x) µg/mL**	**CAL 1 (25x) µg/mL**
Diesel (DRO) o-terphenyl (surrogate)	5000 500	2500 250	1000 100	500 50	200 25
Diesel (DRO)+Motor Oil (ORO) o-terphenyl (surrogate)	5000 each 500	2500 each 250	1000 each 100	500 each 50	200 each 25
JP 4 o-terphenyl (surrogate)	5000 500	2500 250	1000 100	500 50	200 25
JP 8 o-terphenyl (surrogate)	5000 500	2500 250	1000 100	500 50	200 25

7.8. Initial Calibration Verification (**SCV**) Standard

7.8.1 The initial DRO calibration must be verified using a second source standard. This is prepared from a source other than that used to prepare

the calibration standards and contains DRO at a concentration of 2500 µg/**mL**

7.8.2 Prepare the check standard by adding 0.50 mL of **NSI** Diesel Fuel#2 Standard and 0.25 mL of **NSI o-terphenyl** surrogate stock solution to a 10 mL volumetric flask containing about 3 mL of methylene chloride. Dilute to volume with methylene chloride. Analyze undiluted. Prepare this standard every 6 months.

7.9 Continuing Calibration Verification Standard (CCV)

7.9.1 The continuing calibration standard (**CAL4**) is the mid-point of the calibration curve and is prepared daily. See Section 7.7.1.

7.10 The concentrations of surrogate and spiking solutions added to the QC samples are given in the following table (for specific preparation and addition instructions of these standards see SOP 2.2.3.1 and SOP 2.2.3.8):

Parameter	Matrix	LCS	Matrix Spike	Surrogate
DRO 8015	Water	2.5 mg/L	2.5 mg/L	0.25 mg/L
TN DRO	Water	1 mg/L	1 mg/L	0.25 mg/L
JP4	Water	0.5 mg/L	0.5 mg/L	0.25 mg/L
JP8	Water	0.5 mg/L	0.5 mg/L	0.25 mg/L
Total Jet Fuel	Water	0.5 mg/L	16 mg/L	0.25 mg/L
DRO 8015	Soil	125 mg/Kg	125 mg/Kg	12.5 mg/Kg
TN DRO	Soil	20 mg/Kg	20 mg/Kg	10 mg/Kg
JP4	Soil	12.5 mg/Kg	12.5 mg/Kg	12.5 mg/Kg
JP8	Soil	12.5 mg/Kg	12.5 mg/Kg	12.5 mg/Kg
Total Jet Fuel	Soil	12.5 mg/Kg	12.5 mg/Kg	12.5 mg/Kg

8.0 Sample Preservation and Storage

8.1 Sample extracts are stored at 2 – 6 °C until analysis.

8.2 Soil samples must be extracted within 14 days of collection, and extracts must be analyzed within 40 days of extraction.

8.3 Water samples must be extracted within 7 days of collection. Extracts must be analyzed within 40 days of extraction.

8.4 Details of the preservation and handling of samples are found in the tables in Sample Control SOP 4.1, “Receiving Samples” and 4.6, “Storing Samples.”

9.0 Quality Control

9.1 Method Blank

9.1.1 The method blank consists of a matrix similar to that of its associated samples that is free from the analytes of interest. The method blank is processed with the same procedures under the same conditions as the associated samples. A method blank is prepared with each extraction batch of up to 20 field samples. For the matrix used and preparation instructions for the method blank, refer to the associated sample preparation procedures.

9.1.2 Any petroleum hydrocarbon present in the blank must be less than the reporting limit.

9.1.2.1 To meet the requirements of the **DoD**-QSM, the blank must contain less than one-half the reporting limit of any petroleum hydrocarbon.

9.1.3 If the criteria in Section 9.1.2 are not met**,** instrument maintenance may be necessary, see **Section** 9.10. Reanalyze the blank, if contamination persists the entire batch must be re-extracted and re-analyzed.

9.1.4 Any analyte detected in the method blank at a concentration between the reporting limit and the MDL is reported as an estimated concentration.

9.1.5 Any analyte detected in the method blank and the associated samples is flagged with a "B" flag on the report form and explained in the SDG narrative.

9.1.6 For project specific QC sample requirements refer to the LIMS.

9.2 Laboratory Control Sample (LCS)

9.2.1 The LCS is a matrix free from the analytes of interest spiked with verified known amounts of analytes. An LCS is prepared with each extraction batch of up to 20 samples. For the matrix used and preparation instructions for the LCS, refer to the associated sample preparation procedures.

9.2.2 If the LCS fails, the entire batch must be re-extracted if sample volume permits. If additional sample volume is not sufficient to permit re-

extraction or if corrective action, such as instrument maintenance or re-extraction with or without new standard preparation, fails and data associated with an LCS which fails acceptance criteria are reported the data are qualified in the SDG narrative. Reference the "Q" flag for data reported to meet the requirements of the **DoD**-QSM.

9.2.2.1 Precision and accuracy criteria for the LCS/LCSD are given below.

Parameter	RPD	% Recovery	RPD	% Recovery
	Water		Soil	
DRO (8015**C**)	25	50-139	25	50-150
ORO and TN DRO	40	50-139	40	50-150
JP4	20	40-120	20	40-120
JP8	20	40-120	20	40-120

9.2.2.2 If the LCS fails high and the associated samples are non-detects, do not re-extract. Report the data and narrate the failing LCS in the case narrative.

9.3 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

9.3.1 An MS/MSD pair is prepared for every SDG (if requested by the client) when the client supplies sufficient sample volume.

9.3.2 Precision and accuracy criteria for the MS/MSD are given below. Matrix interference can be confirmed through evaluation of the LCS.

Parameter	RPD	% Recovery	RPD	% Recovery
	Water		Soil	
DRO (8015**C**)	25	50-150	25	50-150
ORO and TN DRO	25	50-150	25	50-150
JP4	20	40-120	20	40-120
JP8	20	40-120	20	40-120

9.3.3 If the MS/MSD fails criteria and the LCS passes, the data are to be reported with the failure attributed to the sample matrix and discussed in the SDG narrative.

9.3.4 To meet the requirements of the **DoD**-QSM, the MS/MSD spike recoveries must be assessed using the LCS recovery limits in Section 9.2.2 and the RPD criteria of ≤ 30 %.

9.3.4.1 When reporting MS/MSD data which do not meet acceptance criteria, any analyte reported in the original un-spiked sample must be qualified as an estimated concentration in the SDG narrative. Refer to the **DoD**-QSM "J" flag.

9.4 Surrogates

9.4.1 All client and QC samples are spiked with o-terphenyl as the surrogate.

9.4.2 Surrogate recovery criteria are 47 to 112% for water and 30 to 127% for soil samples.

9.4.3 If the surrogate fails in a field or QC sample, including the preparation blank and LCS, re-analyze the sample neat (undiluted) unless dilution is necessary to bring target analytes into the calibration range or there is obvious matrix interference in field samples. If the surrogate failure occurs in the reanalysis of a sample, re-extract the sample. If the surrogate failure occurs in the reanalysis of the preparation blank or LCS, the entire batch must be re-extracted. New standard preparation may be necessary. If the surrogate fails in an MS and/or MSD and is consistent with the native sample or the other MS/MSD the failure is attributed to the sample matrix and discussed in the SDG narrative. If the surrogate fails in an MS or MSD and is not consistent with the native sample contact the project manager for guidance; re-extraction may be required if sufficient sample volume remains.

9.4.3.1 To meet the requirements of the **DoD**-QSM, if the surrogate recovery control limits in Section 9.4.2 are not met and sufficient volume remains, re-extract and reanalyze the sample. If obvious chromatographic interference with the surrogate is present, contact the project manager for guidance; re-extraction may not be necessary.

9.4.3.2 If field sample results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "J" flag.

9.4.3.3 If QC samples results are reported with failing surrogate recoveries, qualify the results as estimated concentrations in the narrative. Refer to the DoD-QSM "Q" flag.

9.5 Samples following a high-level sample, containing total petroleum hydrocarbons at levels exceeding the calibration range, must be reanalyzed when an amount is detected above the reporting limit.

9.6 Retention Time Window Study

9.6.1 A retention time window study is conducted over a 72-hour period. Three analyses of the Retention Time Marker Standard (RTMARK) are performed and the mean and standard deviation of the retention time of the first eluting component, of interest (e.g., C_{10} in DRO and C_{12} on TN DRO and the last eluting component, of interest (e.g., C_{28} in DRO and C_{40} in TN DRO) are calculated. The study is performed once per year or after major instrumentation maintenance such as column change occurs for each project specific carbon range, for example, C_{10} to C_{28} for DRO and C_{12} to C_{40} for TN DRO.

9.6.2 The retention time window is established, prior to performing initial calibrations and daily calibration verifications. Subtract 3 standard deviations of the mean retention time (determined from the current 72 hour retention time window study) from the elution time of the first component of interest (e.g., C_{10} in DRO and C_{12} on TN DRO) in the retention time marker standard and add 3 standards deviations of the mean retention time to the elution time of the last component of interest (e.g., C_{28} in DRO and C_{40} in TN DRO) in the retention time marker standard. The data associated with the analysis of the retention time marker are provided in the report.

9.6.2.1 To meet the requirements of the DOD-QSM, the retention time window is established using the mid-point standard in the initial calibration curve or the CCV analyzed at the beginning of the analytical sequence.

9.6.3 The retention time window derived in Section 9.6.2 is applied to the analysis of the continuing calibration verification standard and all subsequent field and QC sample analyses during the analytical sequence.

9.7 Solvent Blank: A solvent blank, consisting of only methylene chloride, is run to assess column bleed. One or more solvent blanks must be run first in any sequence, prior to any standards or instrument blanks. Refer to the section on

calculations for details of the quantitation routine involving background subtraction of the solvent blank.

Note: To verify the solvent blank used for background subtraction is only accounting for column bleed, it must meet instrument blank contamination criteria.

9.8 An instrument blank **(IBLK)** is run after the solvent blank and anytime carryover is suspected to be likely. Analytes detected in the instrument blank must be less than the reporting limit. If not, then another instrument blank must be analyzed, or any sample following an instrument blank that contains analyte above the reporting limit must be reanalyzed.

9.8.1 To meet the requirements of the DOD-QSM, the blank must contain less than one-half the reporting limit of any petroleum hydrocarbon.

9.9 Instrument Maintenance and Trouble Shooting

9.9.1 When linearity is difficult to achieve, verify that the appropriate length of column is inserted in the detector. Examine all column ends and determine if the ends of the columns need to be trimmed.

9.9.2 When the instrument blanks fail, examine the chromatography to determine if there is contamination in the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the contamination is such that baking for 1 hour does not improve the baseline, it may be necessary to change the liner.

9.9.3 When CCV standards fail recovery low, verify that the correct peaks are being named. Also verify that the syringe is not plugged and change the septa. If the CCV standard fails again, examine the chromatography to determine if there is a problem with the baseline that is causing the failure. If so, bake the column for 1 hour or less and run 1-2 methylene chloride blanks to see if this can be corrected. If the calibration verification continues to fail, the instrument will need a new calibration curve.

9.9.4 When CCV standards fail recovery high, verify that the correct peaks are being named. Examine the chromatography to determine if there is contamination on the column that is causing the failure. If so, bake the column for 1 hour or less and run solvents to see if this can be corrected. If the CCV standard fails again, the instrument will need a new calibration curve.

9.9.5 When the CCV standard fails due to drift, change the septum and verify that all the column fittings are secure. Also, determine if there is contamination on the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the CCV standard still fails, it is permissible to update retention times once per 24 hours period. Record in the instrument run log when the retention times are updated.

9.9.6 Common maintenance procedures employed as corrective action include cleaning the injection port, replacing the liner, replacing the septum, replacing the syringe and replacing the column. Other corrective actions include removal of 0.5 meters of the detector end of the columns when linearity is difficult to achieve.

9.9.7 All preventive and routine maintenance as mentioned above is recorded in the instrument run log (Attachment 2). Major maintenance is recorded in the maintenance log (Attachment 4).

9.10 Contingency

9.10.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.10.2 **If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.**

9.10.3 **Any other issues that potentially effect data quality should also be addressed with the Project Manager.**

10.0 Calibration and Standardization

Standards and samples are introduced into the instrument using an autosampler. The calibration curve is established by dividing the total area of the diesel pattern, by the mass injected for five different concentrations of each standard. An injection volume of 1 µL is used for all calibration standards, samples, and quality control samples. In the event of column replacement, a new five point initial calibration must be analyzed.

10.1 Initial Calibration:

10.1.1 Average Response Factor: the maximum %RSD between the initial calibration standard levels is ≤ 20% (25% for TN DRO). Samples must not be analyzed until an acceptable initial calibration has been achieved. Samples must be diluted when a result exceeds the upper calibration standard. Results below the lowest standard are reported down to the MDL and are qualified as estimated concentrations.

10.1.2 Linear Regression: if the %RSD of the calibration factors is > 20% (25% for TN DRO), a least squares regression of the calibration curve must be used. If the correlation coefficient of the calibration curve is ≥ 0.995, the initial calibration is acceptable and is used to calculate analyte concentration. The intercept of the calibration curve must not be forced through the origin, but must be calculated using the five data points.

10.1.3 If the initial calibration fails the entire initial calibration is re-analyzed, instrument maintenance and or a new set of standards may be needed.

10.1.4 Initial Calibration Verification (**SCV**): Once an acceptable initial calibration has been established, analyze a second source initial calibration verification (**SCV**) standard. The analyte recovery in the **SCV** must be ± 20% of that in the initial calibration. If the **SCV** fails acceptance criteria, correct the problem and re-analyze. If the second analysis of the **SCV** fails, analyze a new initial calibration.

10.2 Continuing Calibration Verification (CCV):

10.2.1 A continuing calibration verification (CCV) standard (**CAL4**) must be analyzed at least every 12 hours or every 10 production samples, whichever is more frequent. No more than 20% for 8015C and 25% for TN DRO must be observed between the response of the CCV standard and the response from the initial calibration. If the difference is greater than 20% for 8015C and 25% for TN DRO the CCV must be repeated. If upon repeat analysis, the difference is still greater than 20% for 8015C and 25% for TN DRO corrective action must be taken and a new initial calibration must be established.

10.2.2 Samples must be bracketed by passing calibration verification standards. If an ending calibration verification standard fails, all samples analyzed since the last acceptable CCV must be reanalyzed. **However, if a "closing" standard, which is injected after a group of samples, contains target analytes responses that are great than 20%D, and the**

analyte was not detected in that group of samples, the samples do not require re-injection.

11.0 Procedure

11.1 Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures." **The analyst must complete the instrument run log (Attachment 2). Condition codes are used to document failed and qualified injections (Attachment 5).**

11.2 Refer to Quality Control SOP 13.18, "Manual Chromatographic Peak Integration Procedures" for guidance in performing manual integrations.

11.3 One or more solvent blanks must be run first in any sequence, prior to any standards or instrument blanks. Refer to the section on calculations for details of the quantitation routine involving background subtraction of the solvent blank.

11.4 Retention time windows are set to include the carbon range of interest, for example, C_{10} to C_{28} for DRO or C_{12} to C_{40} for TN DRO. The window is set annually, or whenever instrument conditions change, by a 72 hour retention time study using the Restek DRO Mix (EPA/WISC) or NSI Florida TPH Mix. The retention time window may be updated, if necessary, once in a 24 hour period prior to sample analysis.

11.5 A Continuing Calibration Verification is performed, unless an initial calibration is necessary. The calibration type is the external calibration method utilizing the lump sum of the area of all the peaks within the carbon ranges specified in Section 3.6 for DRO. Refer to the section on calculations for details of the quantitation routine. Typical analytical sequences are outlined in Attachment 1. The analyst sets up the autosampler accordingly. The autosampler is programmed to deliver a 1 µL injection volume.

11.6 **A sample of the** gas chromatographic operating conditions are as follows:

Carrier Gas:	Helium
Flow Rate:	5 - 10 mL/min.
Initial Column Temperature:	60 °C for 3 minutes
Ramp Rate:	12 °C/min.
Intermediate Column Temperature:	250 °C
Intermediate Time:	5 minutes
Final Column Temperature:	300 °C
Ramp Rate:	12 °C/min.
Final Hold time:	4 minutes

Injector Temperature:	250 °C
Detector Temperature:	300 °C

The gas chromatographic operating conditions may change as needed to meet requirements.

11.7 The analyst must complete the information fields in the instrument **run log** (Attachment 2).

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction."

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\,100$$

12.3.2 Matrix spikes:

$$\%\,\mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\,100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.6 Linear Calibration using Least Squares Regression

General linear equation: $y = ax + b$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Mass of the analyte in the calibration standard aliquot injected
b = y-intercept

Linear Regression by Least Squares:

$$y_1^{'} = ax_i + b$$

Where: b = intercept
x_i = Mass of the analyte in the ith calibration standard aliquot injected
$y_1^{'}$ = calculated response for the ith calibration standard

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{N}(y_i - y_1^{'})^2$$

12.7 Correlation Coefficient r:

$$r = \frac{N\left[\sum_{i=1}^{N}(x_i y_i)\right] - \left[\sum_{i=1}^{N}(x_i)\right]\left[\sum_{i=1}^{N}(y_i)\right]}{\sqrt{N\left[\sum_{i=1}^{N}(x_i^2)\right] - \left[\sum_{i=1}^{N}(x_i)\right]^2} * \sqrt{N\left[\sum_{i=1}^{N}(y_i^2)\right] - \left[\sum_{i=1}^{N}(y_i)\right]^2}}$$

Where: r = Correlation Coefficient
N = number of data points (equals 5 in a 5 point curve)
y = response
i = index variable (first data point i = 1)
x = Mass of the analyte in the calibration standard aliquot injected

12.8 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \ x\ 100$$

12.9 Calibration factors for GC analysis are calculated by dividing the total peak area for each chosen component in the standard by the total mass injected (in nanograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.10 Concentration

12.10.1 Quantitation of positive results in samples is accomplished by obtaining a processed file that contains the sum of all the individual peaks and areas in the diesel **range organics** pattern and comparing that to the mean calibration factor of the initial calibration. Quantitation of Diesel Range Organics (DRO) or extractable total petroleum hydrocarbon as Diesel is performed using the external standard technique. The concentration of DRO/extractable TPH as Diesel in samples is determined from summing the total response within the retention time range established dividing it by the average calibration factor. Soils are reported on a dry weight basis. See concentration calculations in Sections 12.10.4 - 12.10.7.

The chromatographic conditions employed may result in column-bleed, producing a rise in the baseline. For this reason, the column-bleed needs to be subtracted from the area of the DRO chromatogram. To do this, a solvent blank (methylene chloride) is analyzed during every 12 hours or every 10 production samples, whichever is more frequent. The area is measured in the same manner as DRO in samples and is subtracted from the area measured for samples and the instrument blank. If more than one solvent blank is analyzed the last one run prior the instrument blank is used for background subtraction.

12.10.2 Samples with results above the upper calibration standard must be diluted to achieve a result within the upper half of the calibration range.

12.10.3 Report the presence of significant peaks outside the chromatographic window. Peaks are significant if they can be distinguished above the noise in a chromatogram. Any peak three times the standard deviation of the signal to noise ratio is statistically significant.

12.10.4 Concentration of aqueous samples for GC analysis using mean calibration factor from the initial calibration:

$$ug/L = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Vo)}$$

where: Ax = area response for the analyte
Vt = volume of the concentrated extract (µL)
Df = dilution factor. If no dilution, Df = 1.0
$\overline{CF}$ = mean calibration factor from the initial calibration
Vo = volume of water sample extracted (mL)
Vi = volume of extract injected (µL)

12.10.5 Concentration of soil samples (dry weight basis) for GC analysis mean calibration factor from the initial calibration:

$$ug/kg = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Ws)(D)}$$

where: Ws = weight of sample extracted, in grams
D (dry weight) = 100 - % moisture/100
Ax, Vt, Df, $\overline{CF}$, Vi have the same definitions as for water.

12.10.6 Concentration of aqueous samples for GC analysis using linear regression:

$$ug/L = \frac{(a(Ax)+b)(Vt)(Df)}{(Vi)(Vo)}$$

where: Ax = area response for the analyte
Vt = volume of the concentrated extract µL)
Df = dilution factor. If no dilution, Df = 1.0
a = slope
b = y-intercept
Vo = volume of water sample extracted (mL)
Vi = volume of extract injected (µ**L)**

12.10.7 Concentration of soil samples for GC analysis using linear regression:

$$ug/kg = \frac{(a(Ax)+b)(Vt)(Df)}{(Vi)(Ws)(D)}$$

where: Ws = weight of sample extracted, in grams
D (dry weight) = 100 - % moisture/100
Ax = area response for the analyte
Vt = volume of the concentrated extract (µL)
Df = dilution factor. If no dilution, Df = 1.0
a = slope
b = y-intercept
Vo = volume of water sample extracted (mL)
Vi = volume of extract injected (µL)

12.11 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. **For example, a 10x dilution is performed using 100 µL sample plus 900 µL diluents for a total volume of 1000 µL. It is recorded on the run log as "10x (100 µL in 1000 µL or 100 µL in 1 mL)."** The dilution must be discussed in the SDG narrative.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Non-Halogenated Organics by GC/FID, SW846 3rd Edition, Update **IV**, **Method 8015C, February 2007**

16.2 "Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID", TN DEC, March 1999

16.3 **TNI** Standards, effective July **2011**, plus revisions

16. 4 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.5 Sample Control SOP 4.6, "Storing Samples"

16.6 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.**2**, **October 25, 2011**

16.7 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.8 Quality Control SOP 13.4, "Numerical Data Reduction"

16.9 Determinative Chromatographic Separations, SW-846 3rd Edition, Update IV, March, 2003, Method 8000C

16.**10** Quality Control SOP 13.6, "Manual Chromatographic Peak Integration Procedures"

17.0 Attachments as Tables, Diagrams**,** and Flowcharts

17.1 Attachment 1 – Example of 8015**C** Analytical Sequences

17.2 Attachment 2 – Example Instrument Run Log

17.3 Attachment 3 - Diesel **Range Organics** Pattern

17.4 Attachment 4 – Example Instrument Maintenance Log

17.5 Attachment 5 – Instrument Log Condition Codes

Attachment 1

Typical Analytical Sequences

Initial calibration	$MeCl_2$ (solvent blank) $MeCl_2$ Instrument Blank RTMARKXY (RT window standard) DRO1XY (calibration standard) DRO2XY DRO3XY DRO4XY DRO5XY CHKSTDXY (calibration verification standard)
Continuing Calibration	$MeCl_2$ $MeCl_2$ Instrument Blank RTMARKXZ (RT window standard) DROMXZ (calibration verification standard) (≤ 10 SAMPLES) $MeCl_2$ $MeCl_2$ RTMARKXA DROMXA (calibration verification standard)

Attachment 2

COMPUCHEM a Division of Liberty Analytical Corp
Diesel Range/Oil Range Run Log: LOGBOOK 4 E (5) 8

Instrument ID ____59_____ File Path: _____ DATE ___/___/___
Amount Injected = 1 µL Method (circle one): 8015B 8015C TN DEC

☐Changed septum ☐Changed liner ☐Baked column ☐Trimmed column

	REJECTED	FILE NUMBER	REPORTED	DATE	COMPUCHEM #	CASE/SDG#	STANDARD ID	CHEMIST	COMMENTS
1				/ /					
2				/ /					
3				/ /					
4				/ /					
5				/ /					
6				/ /					
7				/ /					
8				/ /					
9				/ /					
10				/ /					
11				/ /					
12				/ /					
13				/ /					
14				/ /					
15				/ /					
16				/ /					
17				/ /					
18				/ /					
19				/ /					
20				/ /					

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

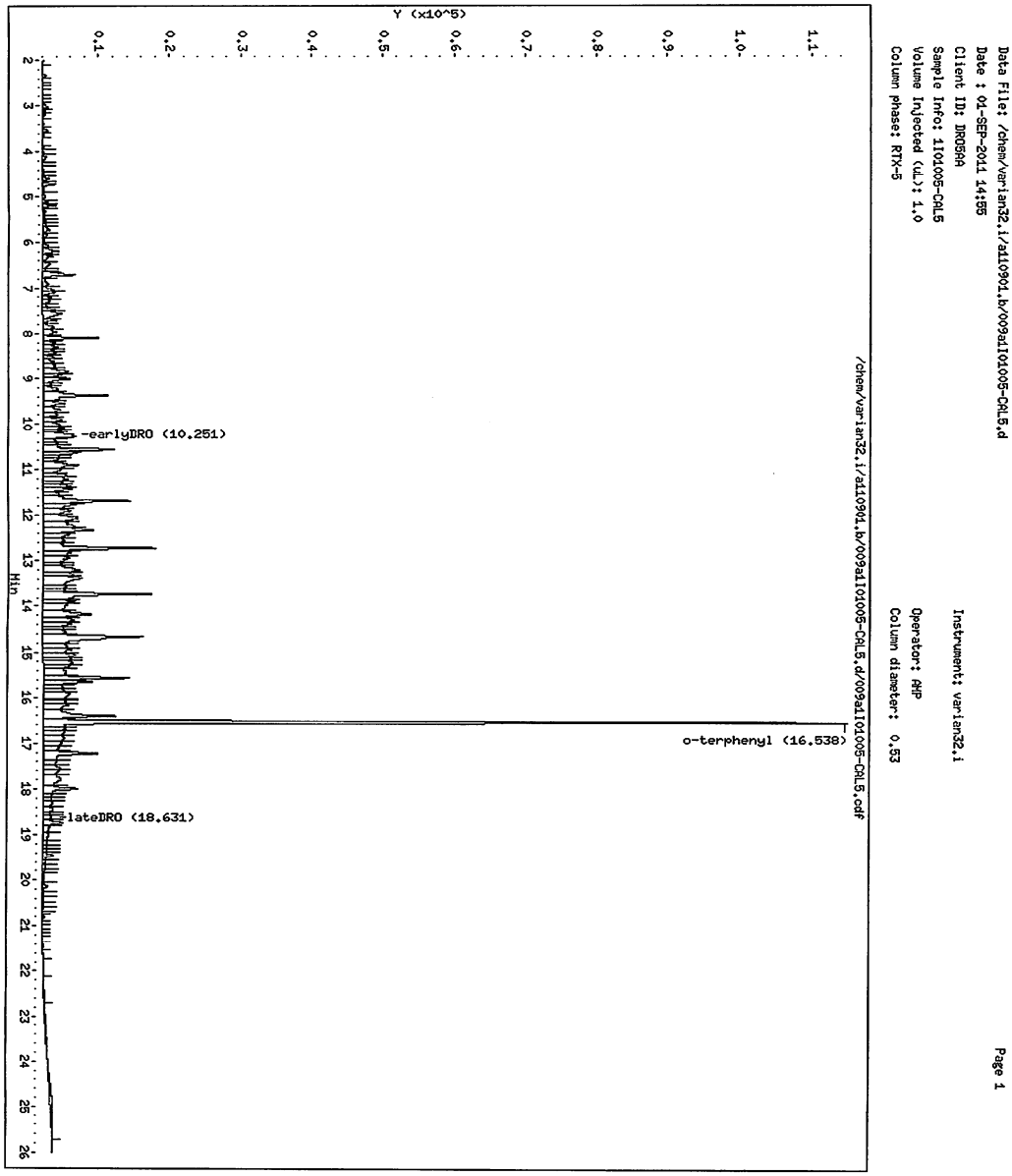
Calibration ID: _____ Sequence ID: _____ Hexane Lot No_____

REVIEWED BY: _____ DATE _____

03/09/09:JR

Note: This page subject to change without notice.

Attachment 3



Note: This page subject to change without notice.

Attachment 4

CompuChem a division of Liberty Analytical Corp. Logbook 4 P(5) 1

GC Extractables Instrument Maintenance Log: 32/33

Date: _____ Time: _____

System: _____

Problem: _____

Action Taken: _____

Parts used:

Part Number	Description	Quantity

File Name Indicating Instrument In Control: _____

Performed By: _____ Date: _____

Reviewed By: _____ Date: _____

11/4/03:dce 00020

Note: This page subject to change without notice.

Attachment 5

Instrument Condition Codes

Code	Explaination
CS	Carry over Suspected from prior analysis
CT	Contamination verified
DI	Requires Dilution
DW	Diluition Wrong
JS	Reinjection same as prior run
LA	Lab Accident (explain in comments)
OT	Other (explain in coments)
OW	Original wrong for QC sample
RP	Reportable prior run
RT	Retention time shift
RU	Repeated Unnecessarily
SF	Spike recoveries failed (high or low)
SI	Spiked inadvertently
SH	Surrogate recoveries high
SL	Surrogate recoveries low
SM	Surrogate or spike missing
IF	Instrument failure
US	Unacceptable Standard (explain in comments)
VR	Verify results
NM	Did not match prior run or duplicate
RN	Reanalyze neat, was run at a dilution
PC	Poor chromatography
SC	Sulfur clean up required
WS	Wrong standard (SOM PCBs)
MI	Misinjection (bad injection)

Code	Explaination
%R	%RSD failed (Ical)
%B	Breakdown failed
%D	%D (CCV)
RT	Retention time shift
RU	Repeated Unnecessarily
IF	Instrument failure
US	Unacceptable Standard (explain in comments)
PC	Poor chromatography
SC	Sulfur clean up required
LA	Lab Accident (explain in comments)
OT	Other (explain in coments)

Note: This page subject to change without notice.

SOP 2.2.3.8

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501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP SOP Section #: 2.2.3.8 Revision #: 4

SOP Title: Preparation of Soil/Sediment/Sludge Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3550B, Method 3550C, and DRO by TN DEC

Effective date: (QA fills in) 10-25-11

- Procedure prepared by: Judith Dozier Date: 10/21/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 10/24/11
- Reason for change: Annual review, addition of LIMS information
- This procedure meets the requirements of the following approved method references: SW-846 3RD Ed., Update III, 12/96, Method 3550B and Update IV, 2/07 Method 3550C; Method for Determination of Extractable Petroleum Hydrocarbons, TNDEC, 1999; DoD QSM, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure

Preparation of Soil/Sediment/Sludge Samples for Diesel Range Organics (DRO) and Oil Range Organics (ORO) by SW-846 Method 3550B, Method 3550C, and DRO by TN DEC

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1.0 Scope and Application

This Standard Operating Procedure (SOP) is applicable to the preparation of soil, sediment or sludge samples for the analysis of extractable total petroleum hydrocarbons (TPH), including diesel range organics (DRO) and jet fuels, by the GC technique using the procedures taken from the SW-846, and TN DEC methodologies. DRO correspond to the range of alkanes from C_{10} to C_{28} (C_{12} to C_{40} for TN DEC) and covering a boiling point range of approximately 170 ^{o}C to 430 ^{o}C.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

This procedure encompasses both EPA Methods 3550B and 3550C. All samples for SC DHEC must be processed under EPA 3550C. This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

2.0 Summary of Method

A measured weight of sample, 20 grams (25-g for TN DRO), is extracted with methylene chloride using a sonicator. The extract is filtered, dried, and concentrated to a final volume of 1.0-mL prior to analysis by Gas Chromatography with Flame Ionization Detector (GC/FID).

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined form analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the**

MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) – The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is smallest concentration of an analyte that must be present in a sample in order to be detected at 99% confidence level. The false negative rate at the LOD is 1%. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of the measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported as qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**- QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single extraction batch. When a group of up to 20 field samples of a similar matrix are prepared as one extraction batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together.

3.7 Extraction Batch – a group of to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 **DoD**-QSM – Department of Defense Quality Systems Manual

3.10 Ohio VAP – Ohio Environmental Protection Agency Voluntary Action Program

3.11 Diesel fuel – consists primarily of straight-chain hydrocarbons (alkenes and alkanes) ranging in length from C_{10} to C_{28} (C_{12} to C_{40} for TN). Carbon chain length of C_{16} and C_{17} predominate in the mixture, whose composition approximates a bell-shaped curve with C_{16} and C_{17} as the mean. Diesel fuel may also contain some aromatic constituents (depending on the source and refining process), including benzene. They usually account for less than 0.1% of the total product.

3.12 JP-8 (JP meaning Jet Propellant) is a kerosene based jet fuel used by the United States Military. Kerosene is a mixture of chains ranging from C_{12} to C_{15}.

3.13 Marker Vial – a vial of same manufacture and size as sample extract vial containing the volume of liquid desired as a final volume for the sample extract. Marker vials are created using class 'A' pipettes.

3.14 **LIMS – Laboratory Information Management System**

4.0 Interferences

4.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines which may cause misinterpretation of chromatographic data. All of those materials must be demonstrated to be free from interferences under the conditions of analysis by running laboratory method blanks.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the site being sampled.

5.0 Safety

5.1 Proper personal protective equipment including lab coat, safety glasses, and gloves must be worn when performing this procedure.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory. The Chemical Hygiene Plan and MSDS are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 General Lab Equipment

6.1.1 Spatulas

6.1.2 Buchner funnel

6.1.3 Whatman 41 filter paper

6.1.4 Teflon® beaker

6.2 Sonicator

6.3 Kuderna-Danish (K-D) apparatus consisting of the following pieces:

6.3.1 Concentrator tube, 10-mL, graduated (Kontes K-570040-1029 or equivalent)

6.3.2 Evaporative flask, 500-mL, (Kontes K-570001-0500 or equivalent)

6.3.3 Snyder column, three-ball macro, (Kontes K-503000-0121 or equivalent)

6.4 Silicon carbide boiling chips, approximately 10/40 mesh.

6.**4**.1 Prior to use, heat the boiling chips to 400°C for thirty minutes or Soxhlet extract them with methylene chloride.

6.5 Water bath, heated, with concentric ring cover, capable of temperature control ± 2 °C. The bath must be located under a ventilation hood.

6.6 Top-loading balance, capable of weighing accurately to 0.01-g

6.7 Vials with Teflon®-lined septa and screw cap or crimp tops, 1.8-mL amber

6.8 Graduated Liter jars with lids

6.9 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40 °C. The N-Evap by Organomation Associates, Inc. (South Berlin, MA) or equivalent is suitable.

6.10 Element DataSystem® LIMS by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook(22 F) or Promium Element® LIMS.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2-6 ^{o}C in the sample preparation laboratory or the Organic Standards laboratory when not in use. **Spiking solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.**

Note: **All reagents and standards must be ACS (American Chemical Society) grade or equivalent. Standards may be purchased as certified neat materials or concentrated solutions.** All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in the applicable analytical SOP. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Sodium sulfate, Mallinckrodt anhydrous, reagent grade, heated at 400 °C for four hours, cooled, and stored in a glass bottle.

7.3 Methylene chloride, pesticide grade or equivalent. (Use only from glass bottles.)

7.4 Methanol – pesticide grade or equivalent

7.5 Stock Diesel Spiking Solution – NSI Diesel Fuel #2 (Catalog # UST-145) at a concentration of 50,000-µg/mL. The expiration date for this solution is provided by the manufacturer.

7.6 Prepare the **DRO LCS/MS Spike** by adding 2.5-mL of NSI Diesel Fuel #2 to methanol in a 25-mL volumetric flask. Dilute to volume with methanol. The concentration of the resulting solution is 5000-µg/mL. Prepare this solution every six months.

7.7 Stock Restek Motor Oil Standard (Catalog# 31464) – 50,000 µg/mL motor oil in methylene chloride (or equivalent).

7.8 Prepare the **ORO LCS/MS Spike** by adding 1.0-mL of Stock Restek Motor Oil Standard to methanol in a 10-mL volumetric flask. Dilute to volume with acetone. The concentration of the resulting solution is 5000-µg/mL. Prepare this solution every six months.

7.9 DRO Surrogate Stock Solution – NSI o-Terphenyl (Catalog # W-1341H) at a concentration of 10,000-µg/mL. The expiration date for this solution is provided by the manufacturer.

7.10 Prepare the DRO Surrogate **Spike** by adding 5.0-mL of NSI o-Terphenyl to methanol in a 100-mL volumetric flask. Dilute to volume with methanol. The concentration of the resulting solution is 500-µg/mL. Prepare this solution every six months.

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples are obtained from the Custodian out of cold storage. They must be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler.

8.3 Soil samples must be extracted within 14 days of collection.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared with each extraction batch.

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS) must be prepared with each extraction batch.

9.2.2 Duplicate LCSs must be prepared with each extraction batch for TN DRO.

9.3 Matrix Spike, Matrix Spike Duplicate and Duplicate Samples

9.3.1 A matrix spike (MS) and matrix spike duplicate (MSD) are prepared for each SDG batch .

9.3.1.1 **DoD**-QSM and SC DHEC require a matrix spike (MS) and matrix spike duplicate (MSD) be prepared with each extraction batch.

9.3.2 A duplicate sample may be prepared instead of the MSD, if samples are expected to contain target compounds.

10.0 Calibration and Standardization

10.1 Balance Calibration

10.1.1 Ensure the balance is calibrated for the day prior to weighing samples. Refer to Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The sample preparation technician must complete the extraction worksheet (for example see Attachment 1). Any unused portion of the worksheet must be Z'd out. The laboratory supervisor or his/her designee reviews the completed worksheet for completeness and accuracy, and then signs and the worksheet. The worksheet accompanies the samples to the analytical laboratory. Include on the worksheet the manufacturer and lot number of reagents/solvents used. If samples are to be analyzed for ORO only substitute ORO spiking solution for DRO spiking solution in the following instructions. If samples are to be analyzed for DRO and ORO add ORO spiking solution in addition to DRO spiking solution in the following instructions.

11.1 Rinse all glassware used in this procedure with methylene chloride prior to use. Discard the rinse solvent in the labeled waste container.

11.2 Allow samples to come to room temperature. Mix the sample in the container thoroughly before taking an aliquot. Discard any foreign objects such as sticks, stones, and leaves.

11.3 Weigh out 20.0-g of sample (25.0-g for TN DRO) into a Teflon® beaker. Record the weight for each sample on the extraction worksheet to the nearest 0.1 g. Add **20-**50 g of sodium sulfate and stir until a homogeneous sandy texture is achieved. Add 0.5-mL of the DRO Surrogate **Spike**.

11.3.1 Note: For Method 3550C, the surrogate spike and validation spike (for the LCS and MS/MSD) are added prior to the addition of the sodium sulfate.

11.4 To prepare the matrix spike and matrix spike duplicate (MS/MSD), choose a sample to use for original, unless a sample is designated to use for QC on the extraction worksheet. Weigh out two additional aliquots of the sample into two Teflon® beakers. Add **20-**50 g of sodium sulfate to each sample aliquot and stir until a homogeneous sandy texture is achieved. Add 0.5-mL of DRO Surrogate **Spike** and 0.5-mL of DRO **LCS/MS Spike** to each sample aliquot.

11.4.1 For TN DRO MS/MSD, add 0.1-mL of DRO **LCS/MS Spike** and 0.5-mL of DRO Surrogate **Spike** to each sample aliquot.

11.4.**2 For DRO/ORO MS/MSD, add 0.5-mL of DRO LCS/MS Spike and 0.5-mL of ORO LCS/MS Spike to each sample aliquot.**

Note: For Method 3550C, the surrogate spike and validation spike (for the LCS and MS/MSD) are added prior to the addition of the sodium sulfate. Refer to August 11, 2010 Policy Memo "SOP Modifications

for Method 3550C (Sonication Extractions for Soil/Sediment Samples).

11.5 To prepare the LCS, weigh out 50.0-g of sodium sulfate in a Teflon® beaker. Add 0.5-mL of DRO Surrogate **Spike** and 0.5-mL of DRO **LCS/MS Spike**.

11.5.1 For TN DRO LCS, add 0.1-mL of DRO **LCS/MS Spike** and 0.5-mL of DRO Surrogate **Spike** to each sample aliquot. For TN DRO, a LCSD must also be prepared.

11.5.2 **For DRO/ORO LCS, add 0.5-mL of DRO LCS/MS Spike and 0.5-mL of ORO LCS/MS Spike to each sample aliquot.**

Note: For Method 3550C, the surrogate spike and validation spike (for the LCS and MS/MSD) are added prior to the addition of the sodium sulfate. Refer to August 11, 2010 Policy Memo "SOP Modifications for Method 3550C (Sonication Extractions for Soil/Sediment Samples).

11.6 To prepare the method blank, weigh out 50.0-g of sodium sulfate in a Teflon® beaker and add 0.5-mL of the DRO Surrogate **Spike**.

Note: For Method 3550C, the surrogate spike and validation spike (for the LCS and MS/MSD) are added prior to the addition of the sodium sulfate. Refer to August 11, 2010 Policy Memo "SOP Modifications for Method 3550C (Sonication Extractions for Soil/Sediment Samples).

11.7 Add 100-mL of methylene chloride to all samples.

11.8 Sonicate the samples for 3 minutes with the ¾ inch sonicator horn ½ inch below the surface of the solvent.

11.9 Decant the solvent layer into Buchner funnel containing Whatman 41 filter paper**, or equivalent**. Filter into a 500-mL sidearm flask using a vacuum.

11.10 Repeat the extraction twice more using 100-mL aliquots of methylene chloride. Before each extraction, thoroughly mix the solid residue, and make certain that the sodium sulfate is free flowing and not a consolidated mass. As needed, break up large lumps with a clean spatula. Decant and filter the extraction solvent after each sonication as above. Collect these extracts in the same flask described in **Section** 11.9.

11.11 Pour the combined extracts into a K-D apparatus. Rinse the beaker with 20 to 30-mL of methylene chloride to complete the transfer.

Note: For Method 3550C, the extract is required to be poured through a powder funnel inserted into the top of the KD apparatus and containing a glass wool plug and 10 g of furnaced sodium sulfate.

11.12 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Wet the Snyder column by adding approximately 1-mL of methylene chloride to the top.

11.13 Place the K-D apparatus on a hot water bath so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. The temperature of the water bath should be at approximately **85** °C or adjusted as required to complete the concentration in 15 to 20 minutes.

11.14 When the apparent volume of extract reaches 4-mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.15 Remove the Snyder column and evaporative flask and place the concentrator tube on the Organomation (nitrogen evaporation apparatus) or use the micro-Snyder technique to concentrate the extract. Concentrate to an apparent volume of 0.3-mL.

11.16 Quantitatively transfer to a 1.8 –mL screw cap or crimp top vial. Pipete the extract into the vial. Rinse the concentrator tube with approximately 0.4-mL of methylene chloride and add the rinseate to the vial. Adjust the final extract volume to 1.0-mL using the 1.0 – mL Marker Vial.

11.17 Attach a label to the vial. The label must contain the following information:

CompuChem Number: XXXXXX
Extraction Procedure Method
Date Extracted: XX/XX/XX

11.18 Deliver the batch of labeled extracts, with the completed extraction worksheet and internal chain-of-custody form, to the GC analytical laboratory. Place the extracts in the designated cold storage unit.

12.0 <u>Data Analysis and Calculations</u>

N/A

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained in electronic form on intranet QA server and in hardcopy form in the QA department or in the data storage room.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3550B.

16.2 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846, 3rd Edition, Update IV, 02/07, Method 3550C

16.3 Leaking Underground Fuel Tank Field Manual: Guidelines for Site Assessment, Cleanup, and Underground Storage Tank Closure. State of California, Leaking Underground Fuel Tank Task Force. May 1988.

16.4 "Method for Determination of Extractable Petroleum Hydrocarbons by GC/FID", TN DEC, March 1999

16.5 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.6 **TNI** Standards, effective July **2011**, plus revisions

16.7 EPA QA-G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.8 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.9 Sample Control SOP 4.6, "Storing Samples"

16.10 Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

16.11 CompuChem Policy Memo "SOP Modifications for Method 3550C (Sonication Extractions for Soil/Sediment Samples)", August 11, 2010

16.12 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010, plus revisions

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example** Extraction Worksheet

Attachment 1

PREPARATION BENCH SHEET

Assigned To _____

Employee ID Number _____

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial	Final	QC	Surr (uL)	Comments

Description	Spike Amount (uL)	Lot Number
SURROGATE		
SPIKE		

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

Manuf. and lot # of reagents/solvents used: _____

(Rev. 0 3/11/2011) Page 1 of 1

Note: This page subject to change without notice.

SOP 2.2.4.1

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Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP SOP Section #: 2.2.4.1 Revision #: 14

SOP Title: Simple Preparation for Pesticide/PCBs in Water by SW-846

Effective date: (QA fills in) 10/31/11

- Procedure prepared by: Judith Mozer Date: 10/27/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 10/28/11
- Reason for change: Annual review; change in vendor for custom mixes
- This procedure meets the requirements of the following approved method references: SW-846 Update III 3rd Ed, 12/96 method 3510C; DoD QSM, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure: Sample Preparation for Pesticides/PCBs in Water by SW-846

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If words above are not highlighted, this is an uncontrolled copy of this document.

Sample Preparation Procedure: Sample Preparation for Pesticides/PCBs in Water by SW-846

1.0 Scope and Application

This Standard Operation Procedure (SOP) describes the preparation of aqueous samples using the separatory funnel technique (Method 3510C) for the analysis of pesticides/PCBs following Method **8081B**/8082**A**.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary

A measured volume of sample, approximately 1 liter, is solvent extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried, exchanged to hexane, and adjusted to a final volume of 5.0 mL. Optional cleanup techniques such as Florisil column cleanup, Method 3620B, may be used

3.0 Definitions

3.1 Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte **(40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL

must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory

control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for the DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.7 Extraction Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 DoD-QSM – Department of Defense Quality Systems Manual

3.10 LIMS Laboratory Information Management System

4.0 Interferences

4.1 Interferences may be caused by contaminants in solvent, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms (GC). All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory reagent blanks.

4.2 Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector (ECD). These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Interference from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.3 Matrix interference may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from source to source, depending on the nature and diversity of the site being sampled. The Florisil column cleanup procedures may be used to overcome such interference in order to achieve the MDLs.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and MSDS are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 Glassware

6.1.1 Separatory funnel, 2000 mL, with Teflon® stopcock

6.1.2 1000 mL graduated cylinders

6.1.3 250 mL Erlenmeyer flask, glass

6.1.4 Concentrator tube, 10 mL, graduated (Kontes K-570050-1025, or equivalent).

6.1.5 Evaporative flask, Kuderna-Danish, 500 mL (Kontes K-570001-0500 or equivalent). Attach to the concentrator tube with springs, rubber bands, or a blue Keck clip.

6.1.6 Snyder column, three-ball macro (Kontes K-503000-0121, or equivalent)

6.1.7 Graduated Glass jars – 1 L

6.2 Pyrex glass wool: Heat in a 400 °C oven for 4 hours before use.

6.3 Powder funnels

6.4 Silicon carbide boiling chips, approximately 10/40 mesh.

6.4.1 Heat to 400 °C for 30 minutes, or Soxhlet extract with methylene chloride for 4 hours.

6.5 Water bath, heated with concentric ring cover, capable of temperature control within ± 2 °C. The bath should be used in a hood.

6.6 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40 °C. The N-Evap by Organomation Associates, Inc. South Berlin, MA (or equivalent) is suitable.

6.7 Wide range pH paper (0-14 pH range).

6.8 Syringes

6.8.1 100 µL, 250 µL, 500 µL, and 1000 µL (1 mL)

6.9 10 mL clear serum glass vials, with Teflon® lined screw caps

6.10 Element DataSystem® LIMS by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook or Promium Element LIMS.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2-6 ^{o}C in the laboratory when not in use. **Spiking solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.**

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in the applicable analytical SOP. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Solvents

7.2.1 Methylene chloride, pesticide grade

7.2.2 Hexane, pesticide grade

7.3 Sodium sulfate, granular, anhydrous

7.3.1 Heat at 400 °C for 4 hours in a shallow tray. Cool and store in glass jars.

7.4 Sodium hydroxide solution (10 N)

7.4.1 Dissolve 40 g of NaOH pellets in reagent water and dilute to 100 mL.

7.5 Sulfuric acid concentrated

7.6 Surrogate Stock Solution

7.6.1 Restek SOM01.1 Pesticide Surrogate Standard solution (Catalog # 32453) containing 2,4,5,6-Tetrachloro-m-xylene (TCX) at 100 µg/mL and Decachlorobiphenyl (DCB) at 200 µg/mL in acetone. The expiration date for this solution is provided by the manufacturer.

7.7 **Pest/PCB Surrogate Spike**

7.7.1 Prepare the **Pest/PCB Surrogate Spike** by adding 1.2 mL of Restek SOM01.1 Pesticide Surrogate Standard to acetone in a 200 mL volumetric flask and dilute to volume with acetone. The resulting surrogate solution contains TCX at 0.6 µg/mL and DCB at 1.2 µg/mL. Prepare this solution every six months.

7.8 **8081 Pest LCS/MS Intermediate Stock**

7.8.1 **Prepare the 8081 Pest LCS/MS Intermediate Stock by adding 1 mL NSI 8081A Organochlorine Pesticide** Mix (C-**617**) **to**

methanol in a 10.0 mL volumetric flask. The resulting concentrations are 100 µg/mL **for the compounds. The stock is prepared every 6 months.**

7.8.2 **Prepare the 8081 Pest LCS/MS Spike by adding 0.15 mL of the 8081A Pest LCS/MS Intermediate Stock in methanol in a 50.0 mL volumetric flask. The resulting concentration for compounds is 0.3 µg/mL. Prepare this solution every 6 months.**:

7.8.**3** A matrix spike solution containing Technical Chlordane or Toxaphene is available to clients that request that these compounds be spiked into matrix spikes and laboratory control samples. Technical Chlordane and Toxaphene are spiked separately.

7.8.**4** Custom TCLP Pesticide Spike (**NSI** Custom **TCLP Spike** #Q-4740) is used as the spiking solution for Toxaphene. This solution contains Toxaphene at 10 µg/mL. The expiration date for this solution is provided by the manufacturer.

7.8.**5** Stock Technical Chlordane Standard – Restek Technical Chlordane Standard (Catalog # 32072) at 5000 µg/mL in isooctane. The expiration date for this solution is provided by the manufacturer.

7.8.6.1 Prepare the Technical Chlordane spiking solution by adding 10 µL of Restek Technical Chlordane Standard to acetone in a 50 mL volumetric flask and dilute to volume with acetone. This solution contains Technical Chlordane at a concentration of 1.0 µg/mL. Prepare this solution every six months.

7.9 PCB **LCS/MS Spike**

7.9.1 **NSI 8082** Aroclor Mix **(C-496)** containing both **1016/1260** Aroclors at 1000 µg/mL. The expiration date for this solution is provided by the manufacturer.

7.9.1.1 PCB **LCS/MS** Spik**e**

7.9.1.1.1 Prepare the PCB **LCS/MS Spike** by adding 0.25 mL **NSI 8082 Aroclor Mix** to methanol in a 50 mL volumetric flask. Dilute to volume with methanol. The resulting concentration of the spiking solution

is 5.0 µg/mL. Prepare this solution every six months.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All samples must be extracted within seven days of sample collection. All extracts must be analyzed within 40 days of extraction.

8.3 Prior to analysis, all extracts must be stored under refrigeration at 2 – 6 ^{o}C in the reach-in storage unit in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared with every batch of up to 20 samples.

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS), must be prepared with every batch of up to 20 samples.

9.3 Matrix Spike, Matrix Spike Duplicate, and Duplicate Sample

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) must be prepared for every batch of up to 20 samples.

9.3.2 A duplicate sample may be prepared instead of the MSD, if samples are expected to contain target compounds.

9.4 Contingency

9.4.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.4.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.4.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration and Standardization

NA

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 Rinse all glassware with methylene chloride before use, including the powder funnel containing sodium sulfate used to dry the extract. Do not use any glassware that appears to be dirty or cracked.

11.2 Thoroughly mix the sample before aliquotting. Using a 1 liter graduated cylinder or graduated glass jar, measure out a 1 liter sample aliquot and transfer to a two-liter separatory funnel. Add 0.5 mL of **Pest/PCB S**urrogate **Spike** to the separatory funnel using a volumetric syringe and mix well. Record the sample volume on the extraction worksheet (Attachment 1).

11.3 Check the pH of the sample with wide range paper and adjust to a pH to between 5 and 9 with 10N sodium hydroxide or concentrated sulfuric acid.

11.4 Prepare the method blank by adding 1 liter of reagent water to a separatory funnel. Add 0.5 mL of **Pest/PCB Surrogate Spike**.

11.5 Prepare the duplicate matrix spikes (MS/MSD) following step 11.2 using two aliquots from one original unspiked sample. Add 1.0 mL **Pest LCS/MS Spike** and 0.5 mL of **Pest/PCB Surrogate Spike** to the MS/MSD.

11.6 Prepare a LCS by adding 1 liter of reagent water to a separatory funnel. Add 1.0 mL of **Pest LCS/MS Spike** and 0.5 mL of **Pest/PCB Surrogate Spike**.

11.7 Add 60 mL of methylene chloride to each separatory funnel and extract by shaking the funnel for 2 minutes, with periodic venting under the hood to release excess pressure.

11.8 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third of the volume of the organic solvent, employ mechanical techniques to complete the phase separation. The optimum technique depends on the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical means.

11.8.1 To break an emulsion using centrifugation, drain the emulsion into a centrifuge bottle. Place the bottle in the centrifuge. (Always use a bottle with approximately the sample volume of methylene chloride as a counter-weight, if only one sample is being centrifuged). Secure the lid of the centrifuge and start. Let the sample(s) spin for 2 minutes at 2000 RPM and turn off the centrifuge. Once the sample(s) have stopped spinning, remove the bottles. Pour the sample extract back into the appropriate separatory funnel. Rinse the bottle with 5 to 10 mL of methylene chloride and pour the rinse into the separatory funnel.

11.9 When methylene chloride layer (bottom layer) has been separated from the sample, drain the methylene chloride through a powder funnel containing glass wool and sodium sulfate into the Erlenmeyer flask.

11.10 Add a second 60 mL volume of methylene chloride to each separatory funnel and repeat the extraction, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

11.11 Assemble a 500 mL K-D evaporative flask, 10 mL concentrator tube, and three-ball **micro** Snyder column.

11.12 Pour the combined extract into K-D apparatus. Rinse the Erlenmeyer flask with 20 to 30 of methylene chloride to complete the quantitative transfer.

11.13 Add one or two clean boiling chips to the evaporative flask and attach a three-ball **micro** Snyder column. Wet the Snyder column by adding approximately 1 mL of methylene chloride to the top of the column.

11.14 Place the K-D apparatus on a hot water bath (**~100** °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 - 30 minutes. At the proper rate of distillation, the

balls of the Snyder column will actively chatter, but the chamber will not flood with condensed solvent. When the apparent volume of extract reaches 4 mL, remove the K-D apparatus from the bath. Allow it to drain and cool.

11.15 After the sample has cooled, add 50 mL of hexane to the K-D. Mix the solvent in the K-D apparatus by carefully tilting the K-D and allowing the solvent to flow out of the attached concentrator tube. Gently swirl the K-D to mix the hexane with the extract.

11.16 Wet the top of the Snyder column by adding approximately 1 mL of hexane. Place the K-D apparatus on the hot water bath. When the apparent volume of the extract reaches 4 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

11.17 Remove the Snyder column and then carefully remove the 10 mL concentrator tube.

NOTE: During all concentration steps, the solvent extract must never be allowed to go to dryness.

11.18 Final Concentration technique

11.18.1 Place the concentrator tube in the N-Evap and evaporate the solvent volume to 5.0 mL using a gentle stream of clean, dry nitrogen. Alternately use micro-Snyder concentration.

11.19 Quantitatively transfer the contents of the concentrator tube to a clear 5 mL vial. Label the vial with the CompuChem number, **extraction** procedure code, and date extracted.

11.20 Complete all extraction worksheet, verifying that final volumes are correct. Any unused portion of the worksheet must be "z'd" out. The laboratory supervisor (or his or her designee) reviews the completed worksheet for accuracy and completeness and then signs it. All information fields must be completed including the manufacturer and lot number of reagents/solvents used.

11.21 Deliver the worksheet and sample extracts to the analytical laboratory, under chain-of-custody.

11.22 Florisil column cleanup using **SOP 2.6.5** "Manual Florisil Cartridge Cleanup **of Water and Soil Extracts** for **the Analysis of** Pesticide/PCB **by SW-846**" may be performed if indicated by the GC laboratory after initial analysis.

12.0 Data Analysis and Calculations

N/A

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained in electronic form on intranet QA server and in hardcopy form in the QA department or in the data storage room.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3510C

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 **TNI** Standards, effective July **2011**, plus revisions

16.4 EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs), EPA/600/B-07/001, April 2007.

16.5 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.6 Sample Control SOP 4.6, "Storing Samples"

16.7 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example** Extraction Worksheet

Attachment 1

Assigned To _____

Employee ID Number _____

PREPARATION BENCH SHEET

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial	Final	GPC	QC	Surr (uL)	Comments

Description	Spike Amount (uL)	Lot Number
SURROGATE		
SPIKE		

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

Reviewed By: _____

Manuf. and lot # of reagents/solvents used: _____

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Notice: This page subject to change without notice.

SOP 2.2.4.2

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure ____ revised procedure ✓ outdated procedure (archive) ____

- Procedure Code: SPP-~~733~~ -169 5/4/2011 SOP Section #: 2.2.4.2 Revision #: 13

SOP Title: Low Level Preparation for Analysis of Pesticides/PCBs in Soil/Sediment/Sludge by SW-846 Methods 3550B and 3550C

Effective date: (QA fills in) 5/6/11

- Procedure prepared by: J-Rdp Date: 04/29/2011
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Miller Date: 05-06-2011
- Reason for change: Addition of DoD-QSM protocols and procedures, Addition of LIMS information; Change in spiking information
- This procedure meets the requirements of the following approved method references: SW-846 Update III, 3rd Ed. Method 3550B, 12/96; Update IV 3rd Ed. Method 3550C, 2/07; DoD-QSM Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure –**169** Low Level Preparation for Analysis of Pesticides/PCBs in Soil/Sediment/Sludge by SW-846 **Methods 3550B and 3550C**

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If words above are not highlighted, this is an uncontrolled copy of this document.

Sample Preparation Procedure –**169** Low Level Preparation for Analysis of Pesticides/PCBs in Soil/Sediment/Sludge by SW-846 **Methods 3550B and 3550C**

1.0 Scope and Application

The following sample preparation procedure is designed to prepare soil/sediment/sludge samples for pesticide and Aroclor analysis by GC/ECD. The soil samples are extracted by sonication. GPC and Florisil are optional cleanup procedures.

Note: This procedure is not used for the extraction of PCBs under the Ohio VAP. PCB-only SOPs are used.

Staff members performing the procedures described in this Standard Operating Procedure (SOP) are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A 30 g aliquot of sample is spiked with the surrogate solution and then mixed with sodium sulfate and extracted by sonication with a 1:1 methylene chloride:acetone solvent mixture**.** The extract is then filtered and concentrated by Kuderna-Danish (K-D) evaporative flask. Gel permeation chromatography (GPC) cleanup, Method 3640A following SOP 2.6.1, "Gel Permeation Chromatography (GPC) Cleanup of Sol**id** and **Aqueous** Extracts for **Semivolatile Organic Compounds** Pesticides**, and Polychlorinated Biphenyls"** may be performed **prior to hexane exchange**.

Florisil column cleanup, Method 3620B, is another optional cleanup procedure performed using SOP 2.6.5, "Manual Florisil Cartridge Cleanup of Water and Soil Extracts for the Analysis of Pesticide/PCB by SW-846"**.**

This procedure encompasses both EPA Methods 3550B and 3550C. All samples for SC DHEC must be processed under EPA 3550C. This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.). **The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.4 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.5 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.6 Reporting Limit (RL) –

3.6.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.6.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 **DoD**-QSM – Department of Defense Quality Systems Manual

3.10 LIMS – Laboratory Information Management System

3.11 Extraction Batch – a group of up to 20 field samples of a similar matrix, method specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate prepared together at the same time.

4.0 Interferences

4.1 Method interferences might be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware. These contaminants lead to discrete artifacts or to elevated baselines in gas chromatography (GC). Routinely, all of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running reagent blanks prepared with each batch.

4.2 Interferences caused by phthalate esters can pose a major problem in pesticide analysis. Common flexible plastics contain varying amounts of phthalates which are easily extracted during laboratory operations, so cross-contamination of glassware frequently occurs when plastics are handled. Interferences from phthalates can best be minimized by avoiding the use of such plastics in the laboratory.

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending on the nature of the site being sampled. The cleanup procedures must be used to remove such interferences in order to achieve specific method detection limits.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The Chemical Hygiene Plan and the MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Sonic cell disruptor **Branson, model** (**1000**-watt with pulsing capability**). and quad** 3/4-inch **tapered** disrupter horn**s**, or equivalent device with a minimum **300**-watt output capability.

Note: In order to ensure that sufficient energy is transferred to the sample during extraction, the horn must be replaced if the tip begins to erode. Erosion of the tip is evident if it has a rough surface.

6.2 Sonabox (or equivalent) for use with disrupter to decrease cavitation sound

6.3 Teflon® beakers – 250 mL

6.4 Filtration apparatus:

6.4.1 Buchner funnel

6.4.2 Filter paper – **Munktell grade 389, 70mm diameter**

6.4.3 Filtration flask – 500 mL

6.5 Kuderna-Danish (K-D) apparatus

6.5.1 Concentrator tube – 10 mL, graduated (Kontes K-570040-1029, or equivalent)

6.5.2 Evaporative flask - 500 mL (Kontes K-470001-0500, or equivalent)

6.5.3 Snyder column - three-ball macro (Kontes K-503000-0121, or equivalent)

6.6 Silicon carbide boiling chips - approximately 10 to 40 mesh.

6.6.1 Heat the chips to 400 ^{o}C for 30 minutes prior to use. If know heated boiling chips are available, rinse new boiling chips with methylene chloride before use.

6.7 Water bath - heated, with concentric ring cover, capable of temperature control (± 5 °C).

NOTE: Always use the water bath in a hood.

6.8 Top-loading balance - capable of weighing accurately to ± 0.01 g

6.9 Nitrogen evaporation device equipped with a heated bath that can be maintained at 35 °C to 40 °C, N-Evap by Organomation Associates, Inc., South Berlin, MA, or equivalent

6.10 Vials and caps - 2 mL for GC autosampler

6.**11** **4**0 mL **vial** with Teflon®-lined screw-cap for sulfur removal, if needed

6.**12** Spatulas - disposable wooden tongue-depressors

6.**13** Pipets - volumetric 1.0 mL or 2.0 mL (optional)

6.**14** Syringes - 1.0 mL or 2.0 mL (optional)

6.**15** Vials - 10 mL, with screw top and Teflon®-faced seal

6.**16** Tubes - centrifuge, 20- to 15 mL

6.**17** Centrifuge - table top (IEC model Centra-8)

6.18 Vortex mixer

6.**19** Disposable Pasteur glass pipets, 1 mL

6.20 Element DataSystem® LIMS computer software by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook (22F) or Promium Element® LIMS.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2-6 ^{o}C in the laboratory when not in use. **Spiking solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.**

Note: All standards and reagents are subject to change in vendor and in concentration. Standards may be purchased as certified neat materials or concentrated solutions. All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.

7.1 Reagent Water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in the applicable analytical SOP. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Sodium sulfate – Mallinckrodt granular, anhydrous reagent grade, or equivalent

7.2.1 Heat to 400 °C for 4 hours, cool, and store in a glass bottle prior to use.

CAUTION: An open container of sodium sulfate may become contaminated during storage in the laboratory.

7.3 Solvents - pesticide quality or equivalent. Each lot of solvent is tested to demonstrate that it is free of interferences before use.

7.3.1 Methylene chloride

7.3.1.1 Methylene chloride must be certified as acid-free.

7.3.2 Hexane

7.3.3 Acetone

7.4 Surrogate Stock Solution

7.4.1 Restek SOM01.1 Pesticide Surrogate Standard solution (Catalog # 32453) containing 2,4,5,6-Tetrachloro-m-xylene (TCX) at 100 µg/mL and Decachlorobiphenyl (DCB) at 200 µg/mL in acetone.

7.5 Surrogate solution #449

7.4.1 Prepare the surrogate solution by adding 1.2 mL of Restek SOM01.1 Pesticide Surrogate Standard to acetone in a 200 mL volumetric flask and dilute to volume with acetone. The resulting surrogate solution contains TCX at 0.6 µg/mL and DCB at 1.2 µg/mL. Prepare this solution every six months **or sooner if degradation occurs**.

7.6 Pesticide Stock Spiking Solutions

7.6.1 NSI **8081A Organochlorine Pesticide Mix (Catalog# C-617)** containing the following compounds in a 1:1 hexane/toluene solution at **1000** µg/mL:

Compounds	
Aldrin	alpha-BHC
beta-BHC	Delta-BHC
gamma-BHC (Lindane)	alpha-Chlordane
Gamma-Chlordane	Endosulfan I
Heptachlor	Heptachlor epoxide
4, 4'-DDT	**4, 4'-DDD**

Compounds	
4, 4'-DDE	**Dieldrin**
Endosulfan II	**Endosulfan sulfate**
Endrin	**Endrin aldehyde**
Endrin ketone	**Methoxychlor**

7.6.2 **8081A Intermediate Stock Solution**

7.6.2.1 Prepare the 8081A Intermediate Stock Solution by adding 0.5 mL of NSI 8081A Organochlorine Pesticide Mix to methanol in a 5 mL volumetric flask and dilute to volume with methanol. The resulting concentrations are 100 µg/mL for the compounds in Sections 7.6.1. Prepare this solution every six months or sooner if degradation occurs.

7.6.3 Pesticide Matrix Spiking Solution #4030

7.6.3.1 Prepare the Pesticide Matrix Spiking Solution #4030 by adding 0.**15** mL **8081 Intermediate Stock Solution** to methanol in a **50** mL volumetric flask and dilute to volume with methanol. The resulting concentrations are 0.**3** µg/mL for the compounds in **Section** 7.6.1. Prepare this solution every six months **or sooner if degradation occurs**.

7.6.4 A spik**ing** solution containing Technical Chlordane or Toxaphene is available to clients that request that these compounds be spiked into matrix spikes and laboratory samples. Technical Chlordane and Toxaphene are spiked separately.

7.6.5 Custom NSI TCLP Pesticide Spike (Custom #Q-4740) is used as the spiking solution for Toxaphene. This solution contains Toxaphene at 10 µg/mL. The expiration date for this solution is provided by the manufacturer.

7.6.6 Stock Technical Chlordane Standard – Restek Technical Chlordane Standard (Catalog # 32072) at 5000 µg/mL in isooctane.

7.6.6.1 Prepare the Technical Chlordane spiking solution by adding **0.0**1 **mL** of Restek Technical Chlordane Standard to acetone in a 50 mL volumetric flask and dilute to volume with acetone. **The resulting concentration is 1.0 mL of Technical Chlordane.**

Prepare this solution every six months **or sooner if degradation occurs**.

7.7 PCB Stock Spiking Solution

7.7.1 Restek Aroclor® 1016/1260 Mix (Catalog # 32039) containing both Aroclors at 1000 µg/mL.

7.7.1.1 PCB Spiking Solution #4615

7.7.1.1.1 Prepare the PCB spiking solution #4615 by adding 0.5 mL of Restek Aroclor® 1016/1260 Mix to a **100** mL methanol in a volumetric flask and dilute to volume with methanol. The resulting spiking solution contains Aroclor 1016/1260 at 5.0 µg/mL in methanol. Prepare this solution every six months **or sooner if degradation occurs**.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples must be extracted within 14 days of collection.

8.3 Samples are obtained from the **Sample** Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the **Sample** Custodian and placed in the cooler at 2-6 ^{o}C for long-term storage and disposal.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank is prepared with each extraction batch of up to 20 samples and processed with the associated samples.

9.2 Laboratory Control Sample (LCS)

9.2.1 A laboratory control sample (matrix spike blank for NYSASP) is prepared with each extraction batch of up to 20 samples.

9.3 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

9.3.1 A MS and MSD are prepared with each extraction batch of up to 20 samples.

9.4 Duplicates

9.4.1 Duplicates, at a frequency of 5% for soil samples, are required when processing samples submitted to meet the regulatory requirements of South Carolina DHEC. The duplicate matrix spikes satisfy the duplicate requirement.

10.0 Calibration and Standardization

10.1 Ensure the balance is calibrated for the day **of** use following Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The sample preparation technician must complete the proper extraction **preparation bench sheet** for **Methods 8081 and 8082** (**See example,** Attachment 1) including the manufacturer and lot number of reagents/solvents used. Any unused portion of the **bench sheet** must be **"**Z'd**"** out. The laboratory supervisor or his/her designee reviews the **bench sheet** for completeness and accuracy, and then signs the worksheet. The **bench sheet** accompanies the sample extracts to the analytical laboratory.

11.1 Sample Preparation

11.1.1 Thoroughly mix the sample before aliquotting, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks. Also, decant and discard any standing aqueous phase.

11.2 Sonication Extraction

Note: **Originally, Method 3550C required spiking solutions be added prior to the addition of sodium the sodium sulfate. However, the USEPA issued a memo, dated August 5, 2010, recommending that the spiking solutions are not added prior to the addition of the sodium sulfate.**

(See Policy Memo dated August 11, 2010 for further information and instructions.)

11.2.1 Weigh approximately 30 g (± 0.1 g) of sample into a 250 mL Teflon® beaker and add 60 g of granular, anhydrous sodium sulfate. Record the sample weight to the nearest 0.1 g on the extraction **bench sheet**. Stir the sample and sodium sulfate into a homogeneous sandy texture. **Additional sodium sulfate can be added as needed to achieve the free flowing, sandy texture.**

11.2.2 For a sample to be used for the MS and MSD, weigh out four additional 30 g portions of sample, two for the pesticide MS/MSD and two for the PCB MS/MSD. Record the weights to nearest 0.1 g. Add 60 g of sodium sulfate to each aliquot. Add 1 mL of the pesticide matrix spike solution #4030 to one MS/MSD pair and 1.0 mL PCB spiking solution #4615 to the other MS/MSD pair.

11.2.3 Prepare two LCSs by weighing two 30.0 g aliquots of sodium sulfate and adding 60.0 g more to each. Add 1 mL of pesticide matrix spike solution #4030 to the 1st LCS, and add 1.0 mL of #4615 PCB spike solution to the 2nd LCS.

11.2.4 Prepare the method blank by weighing of 90.0 g of sodium sulfate spiked with the 0.5 mL of the surrogate solution #449. It is carried through the entire analytical procedure.

11.2.5 Add 0.5 mL of surrogate solution #449 to all samples, the **LCS,** MS, **and** MSD by using a volumetric pipet or a syringe.

11.2.6 Immediately add 100 mL of 1:1 methylene chloride:acetone to the sample.

11.2.7 Place the bottom surface of the sonicator probe about 1/2 inch below the surface of the solvent but above the sediment layer.

11.2.8 Sonicate for three minutes using a 3/4-inch horn at **a power output between 60 and** 100% with pulse set on 1 second ON and percent duty cycle knob set at 50%. Do <u>not</u> use a microtip. **The setting is dependent upon the sample matrix. The analyst MUST observe very active mixing of the sample and solvent and must adjust the output accordingly.**

11.2.9 Decant and filter the extracts using Buchner funnel filtration.

11.2.10 Repeat the extraction two more times with additional 100 mL portions of 1:1 methylene chloride:acetone. Before each extraction, thoroughly mix the solid residue, and make certain that the sodium sulfate is free flowing and not a consolidated mass. As needed, break up large lumps with a clean spatula. Decant and filter the extraction solvent after each sonication as above. After the final sonication, pour the entire sample into the funnel and rinse the **Teflon® beaker** and funnel with 20 to 30 mL of methylene chloride.

11.2.10.1 If particulate matter is observed, filter the entire extracted sample again by placing a new filter in the original funnel and pouring the extracted sample through the funnel. Rinse with methylene chloride.

NOTE: Do NOT allow the sample to completely dry. Continued application of the vacuum pump may result in the loss of some analytes.

11.2.11 Pour each extract into a K-D evaporative flask and concentrator tube assembly for concentration.

Note: For Method 3550C, the extract is required to be poured through a powder funnel inserted into the top of the KD apparatus and containing a glass wool plug and 10 g of furnaced sodium sulfate.

11.3 Extract Concentration

11.3.1 Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro-Snyder column. Add about 1 mL of methylene chloride to the top of the Snyder column. Place the K-D apparatus on a hot water bath (80 - 85 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 - 30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. Reduce the volume of liquid to less than 10 mL. Remove the K-D apparatus and allow it to drain and cool for at least 10 minutes.

CAUTION: DO NOT ALLOW THE K-D EVAPORATOR TO GO DRY.

11.3.2 If GPC is to be performed, the following procedure is to be used. If not, proceed to Section 11.4.

11.3.2.1 In order to remove most of the acetone, it is absolutely necessary to further reduce the volume of the extracts to 2.0 mL. This is best accomplished using the nitrogen concentration technique, but can be accomplished using the K-D flask if care is taken during the K-D concentration step. The presence of acetone will cause a dead volume to develop in the GPC column and thus will cause loss of surrogates and analytes during GPC cleanups.

Note: This step has not proven to be critical, thus it may be eliminated unless surrogate recoveries indicate otherwise.

11.3.2.2 Adjust the extract volume to 10.0 mL with methylene chloride. Proceed with GPC cleanup (see SOP 2.6.1).

11.3.2.3 Aliquot 4.0 mL of extract, dilute to 10 mL with methylene chloride and proceed with the GPC process.

11.3.2.4 After GPC cleanup, set up a K-D flask with a 10 mL concentrator tube attached. Pour the methylene chloride extract after GPC cleanup into the K-D. Rinse the extract container with methylene chloride to complete the transfer. Place the three-ball macro Snyder column on the K-D apparatus, add 1 to 2 mL of methylene chloride to the top of the Snyder, and place the apparatus on the hot water bath. Concentrate the extract to approximately 4 mL. Remove the K-D apparatus and allow it to cool. Proceed to the solvent exchange into hexane.

11.4 Solvent Exchange into Hexane and Final Concentration

11.4.1 Momentarily remove the Snyder column, add 60 mL of hexane and a new boiling chip, and reattach the Snyder column. Pre-wet the column by adding about 1 mL of hexane to the top. Concentrate the solvent extract as before. When the apparent volume of liquid reaches 3 to 5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 minutes. Alternatively, micro-Snyder concentration may be used.

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CAUTION: DO NOT ALLOW THE K-D EVAPORATOR TO GO DRY.

11.4.2 Concentrate the GPC extract in hexane to a 1.0 mL final volume using the N-EVAP technique. If GPC was not performed concentrate the extract to 5.0 mL.

11.4.3 Label all extracts with the following information:

CompuChem number	XXXXXX
Procedure Code	**8081/8082**
Date Extracted	XX/XX/XX

Complete all **preparation bench sheets** and deliver it with the extracts to the designated area in the GC instrument laboratory.

12.0 Data Analysis and Calculations

N/A

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained in electronic form on intranet QA server and in hardcopy form in the QA department or in the data storage room.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, “Hazardous Waste Disposal”, regarding laboratory procedures for recycling solvent waste streams.

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15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, **Method 3550B,** 12/96**;** Update IV, Method 3550C, 02/07

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 NELAC Standards, effective July 2003, plus revisions

16.5 EPA QA-G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.6 CompuChem Quality Manual, Revision 13, November 17, 2009, plus revisions

16.7 Sample Control SOP 4.1, "Receiving Samples" & Sample Control SOP 4.6, "Storing Samples"

16.8 Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

16.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **10**/**25**/**2010, plus revisions**

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example** Extraction **Preparation Bench Sheet**

Attachment 1

Assigned To _____

Employee ID Number _____

Matrix: Soil

PREPARATION BENCH SHEET

0102807

GC-8081B 8082A-Pest PCB DOD

Prepared using: GC - EPA 3550B_GC

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial (g)	Final (uL)	GPC (uL)	QC	Surr (uL)	Comments
0102807-BLK1	PBLKFV	Blank					500	
0102807-BS1	PFVLCS	LCS					500	PESTICIDE
0102807-BS2	PFWLCS	LCS					500	PCB
0102807-BSD1	PFVLCSD	LCS Dup					500	PESTICIDE
0102807-BSD2	PFWLCSD	LCS Dup					500	PCB
1010109-01RE1	CA-IDW-CARBON	Sample					500	
1010109-02RE1	CA-IDW-SOIL	Sample					500	

	Description	Spike Amount (uL)	Lot Number
SURROGATE	#449 PEST/ARO SURR	500	
SPIKE	4030 Afcee Spike	1000	LCS/LCSD
SPIKE	#4615 PCB Spike	1000	LCS/LCSD

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

Balance ID: _____

GPC Batch: _____ GPC RUN Date: _____ GPC CAL Date: _____ GPC INST#: _____

Florisil Batch: _____ Florisil RUN Date: _____ Water Florisil Lot #: _____

Manuf. and lot # of reagents/solvents used: _____

(Rev. 0 3/11/2011) Page 1 of 1

Note: This page subject to change without notice.

SOP 2.2.4.3

CompuChem
A Division Of
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501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP SOP Section #: 2.2.4.3 Revision #: 10

SOP Title: Extraction of TCLP Leachates for the Determination of Pesticides/PCBs by SW-846

Effective date: (QA fills in) 10/31/11

- Procedure prepared by: Judith Dozier Date: 10/26/11
- Procedure approved by: (If the manager prepared the SOP, a ~~qualified~~ second party should sign) Date: 10/28/11
- Reason for change: Annual Review
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, Method 3510C; DoD QSM Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

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1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the procedure is used to extract pesticides/PCBs from the aqueous leachate generated using Sample Preparation Procedure, "Toxicity Characteristic Leaching Procedure (TCLP) **by SW-846 Method 1311**".

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

Approximately 200-mL of a previously prepared TCLP leachate are diluted to 1000-mL (a 5:1 dilution), and extracted with methylene chloride using a separatory funnel (or continuous liquid-liquid) technique. The methylene chloride extract is dried, exchanged to hexane, and adjusted to a final volume of 10.0-mL. Optional cleanup techniques may be needed.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Units – **µ**g/L

3.3 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.4 **DoD**-QSM – Department of Defense Quality Systems Manual

3.5 2,4,5,6-Tetrachloro-m-xylene - TCX

3.6 Decachlorobiphenyl - DCB

3.7 Extraction Batch – a group of to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.8 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware cause method interferences that lead to discrete artifacts and/or elevated baselines in gas chromatograms (GC). All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks.

Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector (ECD). These compounds generally appear in the chromatogram as broad eluting peaks. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross-contamination of clean glassware routinely occurs when plastics are handled. Avoiding the use of plastics in the laboratory minimizes interference from phthalates. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.

4.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the site being sampled.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and the MSDS are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 Separatory funnel, 2000-mL, with Teflon® stopcock

6.2 Small powder funnel

6.3 Kuderna-Danish Apparatus

6.3.1 Concentrator tube, 10-mL, graduated (Kontes K-570050-1025 or equivalent). Calibration must be checked at the volumes used in the test.

6.3.2 Evaporative flask, 500-mL (Kontes K-570001-0500 or equivalent). Attach to concentrator tube with springs, rubber bands, or blue Keck clip.

6.3.3 Snyder column - three-ball macro (Kontes K-503000-0121 or equivalent).

6.3.4 2-ball micro distillation Snyder column

6.4 1000-mL graduated cylinders or graduated glass jar

6.5 250-mL Erlenmeyer flask, glass

6.6 Pyrex glass wool. (Prepare by heating in a 500 °C oven for 2-4 hours before use.)

6.7 Silicon carbide boiling chips, approximately 10/40 mesh. (Prepare by heating to 400 °C for 30 minutes or Soxhlet extract with methylene chloride for 4 hours, prior to use.)

6.8 Water bath, heated with concentric ring cover, capable of temperature control to ± 2 °C. The bath should be used in a hood.

6.9 Nitrogen evaporation device equipped with a water bath that can be maintained at 35-40 °C. The N-Evap by Organomation Associates, Inc. South Berlin, MA (or equivalent) is suitable.

6.10 Wide range pH paper (0-14 pH range)

6.11 1.0-mL serological pipettes

6.12 Pasteur pipettes, glass, disposable

6.13 10-mL amber serum glass vial with crimp-top aluminum seal and Teflon®-faced septa

6.14 Element DataSystem® LIMS computer software by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the standards preparation log book or in Promium LIMS.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

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Note: All standards and reagents are subject to change in vendor and in concentration. All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this SOP. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Solvents, pesticide grade or equivalent

7.2.1 Hexane

7.2.2 Methylene chloride

7.3 Sodium sulfate, (ACS grade) granular, anhydrous.

7.3.1 Dry in 400° C oven for 4 hours in a shallow tray.

7.4 Sodium hydroxide solution (10N), ACS grade.

7.4.1 Dissolve 40 g of NaOH pellets in reagent water and dilute to 100-mL.

7.5 Sulfuric acid solution, concentrated

7.6 Stock Standard Solutions

7.6.1 Restek SOM01.1 Pesticide Surrogate Standard (100-µg/mL for **TCX** and 200-µg/mL for DCB), Catalog # 32453

7.7 Working Standard Solutions

7.7.1 **Pest/PCB Surrogate Spike**. The surrogate compound concentrations are 0.6-µg/mL for (TCX) and 1.2-µg/mL for Decachlorobiphenyl (DCB).

7.1.2 Prepare **the Pest/PCB Surrogate Spike** by adding 1.2-mL of Restek SOM01.1 Pesticide Surrogate Standard to acetone in a 200-mL volumetric flask. Dilute to volume with acetone.

7.7.3 **Custom NSI** TCLP **Pest Spike (Q-4740)**. **This** solution is **a**purchased working standard **from** NSI Solutions, Inc. The **Custom NSI TCLP Pest Spike (Q-4740)** solution contains the **listed** compounds and concentrations in methanol **as follows:**

Compound	**µg/mL**
gamma BHC (Lindane**)**	0.3
Heptachlor	0.3
Heptachlor epoxide	0.3
Toxaphene	10.0

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All TCLP leachates must be extracted within 7 days of leachate generation.

8.3 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler.

9.0 Quality Control

9.1 Method blank

9.1.1 A method blank must be prepared with each batch of up to 20 samples extracted.

9.2 Laboratory Control Sample (LCS)

9.2.1 A LCS must be prepared with every batch of up to 20 samples extracted.

9.3 Matrix Spike and Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) must be prepared for batch of not more than 20 samples.

9.4 Contingency

9.4.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.4.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.4.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration and Standardization

NA

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The sample preparation technician must complete the extraction worksheet (Attachment 2). Any unused portions must be Z-d out. The laboratory supervisor or designee reviews the completed worksheet for accuracy and completeness and then signs it. The worksheet accompanies the sample to the analytical laboratory. Include on the worksheet the manufacturer and lot number of the reagent/solvents used.

11.1 Rinse all glassware with methylene chloride before use. Do not use any glassware that appears to be dirty or cracked. Return dirty glassware to the glassware prep area for washing, and dispose of broken glassware in proper receptacle.

11.2 Thoroughly mix the sample before aliquotting. Using a one-liter graduated cylinder or 1-liter graduated glass jar, measure 100-mL of the TCLP leachate. Record the volume on the extraction worksheet. Dilute to 500-mL with reagent water, and transfer to a 2 liter separatory funnel. Pipet 0.25-mL **Pest/PCB Surrogate Spike** into the separatory funnel and mix well. Check the pH of the sample using wide range pH paper and adjust to a pH between 5 and 9 with 10N sodium hydroxide solution or concentrated sulfuric acid.

11.3 For the MS/MSD follow **Section** 11.2 using two additional aliquots from one original unspiked sample. Add 0.5 **mL** of **NSI** TCLP **Pest Spike** solution and 0.25-mL of **Pest/PCB Surrogate Spike** to the MS/MSD.

11.4 Prepare a method blank using 500-mL extracted reagent water. Transfer the aliquot to a 2-liter separatory funnel. Add 0.25-mL of **Pest/PCB Surrogate Spike**.

11.5 Prepare a LCS by measuring 100-mL extraction fluid, diluting to 500-mL with reagent water and transferring to a 2 liter separatory funnel. Add 0.5-mL of **NSI TCLP Pest Spike** solution and 0.25-mL **Pest/PCB Surrogate Spike**.

11.6 To prepare the leachate blank, use 100-mL of the TCLP leachate blank and dilute to 500-mL with reagent water. Add 0.25-mL of **Pest/PCB Surrogate Spike**.

11.7 Add 60-mL of methylene chloride to each separatory funnel and extract the samples by shaking the funnel for two minutes, with periodic venting under the hood to release excess pressure. Allow the methylene chloride layer to separate from the water phase for at least 10 minutes. If the emulsion interface between layers is more than one third of the volume of the solvent layer, the technician must employ mechanical techniques to complete the phase separation. The optimum technique depends on the sample, and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical means.

11.7.1.1 To break an emulsion using centrifugation, drain the emulsion into a centrifuge bottle. Place the bottle in the centrifuge. (Always use a bottle with approximately the sample volume of methylene chloride as a counter-weight, if only one sample is being centrifuged). Secure the lid of the centrifuge and start. Let the sample(s) spin for 2 minutes at 2000 RPM and turn off the centrifuge. Once the sample(s) have stopped spinning, remove the bottles. Pour the sample extract back into the appropriate separatory funnel. Rinse the bottle with 5 to 10-mL of methylene chloride and pour the rinse into the separatory funnel.

11.8 When the methylene chloride layer has separated, drain it through a powder funnel containing sodium sulfate into the Erlenmeyer flask.

11.9 Add a second 60-mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

11.10 Assemble a 500-mL Kuderna-Danish (K-D) evaporative flask, a 10-mL concentrator tube, and a three-ball macro Snyder column. Rinse all glassware with methylene chloride.

11.11 Pour the combined extract into the K-D apparatus. Rinse the Erlenmeyer flask with 20-30-mL of methylene chloride to complete the quantitative transfer.

11.12 Add one or two clean boiling chips to the evaporative flask and attach a three-ball macro Snyder column. Wet the Snyder column by adding about 1.0-mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot

water bath (80 to 90 °C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15-30 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 4-mL, remove the K-D apparatus from the bath. Allow it to drain and cool.

11.13 After the sample has cooled, remove the Snyder column. Add 50-mL hexane. Carefully tilt the K-D until the methylene chloride in the concentrator tube flows back into the hexane in the K-D. Gently swirl the KD to mix the contents. Repeat the mixing step several times. Wet the top of the Snyder column by adding about 1-mL of hexane. Replace the K-D apparatus on the hot water bath. When the apparent volume of liquid reaches 4-mL, remove the K-D apparatus. Allow it to drain and cool for at least ten minutes.

Caution: Never allow the extract to go dry during any concentration step.

11.14 Final Extract Concentration

11.14.1 Nitrogen concentration technique

11.14.1.1 Remove the K-D and Snyder column then place the concentrator tube in the N-EVAP and evaporate the solvent using a gentle stream of clean, dry nitrogen. Concentrate the extract to 3-mL.

11.14.2 Micro Snyder concentration technique

11.14.2.1 Attach a 2-ball micro Snyder column to the concentrator tube. Wet the Snyder by adding about 1-mL of hexane to the top. Concentrate the extract by holding the concentrator tube over the hot water bath. When the extract reached an apparent volume of 3-mL, remove it from the bath. Allow the apparatus to drain and cool.

11.15 Quantitatively transfer the contents of the concentrator tube to a 12-mL clear vial. Adjust the final volume of the extract to 5-mL with hexane. Label the amber vial with a green label containing the following information:

- CompuChem number
- Date extracted

11.16 Complete all paperwork, verifying the final volumes, and obtain supervisor or peer review of paperwork and extracts. Deliver the extracts and the associated paperwork to the designated area within the GC instrumentation laboratory.

11.17 Optional Florisil cleanup may be performed, if indicated by the GC laboratory after initial analysis.

12.0 Data Analysis and Calculations

N/A

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for the Evaluation of Solid Waste: Physical/Chemical Methods, SW-846, 3rd Edition, 12/96, Method 3510C.

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.4 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.5 **TNI** Standards**, effective July 2011, plus revisions**

16.6 EPAQA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.7 CompuChem Quality Manual, Revision **14**, **January 25, 2011, plus revisions**

16.8 Department of Defense Quality Systems Manual of Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example** Preparation Worksheet

Attachment 1

Assigned To _____

Employee ID Number _____

PREPARATION BENCH SHEET

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial	Final	Adjusted pH		QC	Surr (uL)	Comments

	Description	Spike Amount (uL)	Lot Number
SURROGATE			
SPIKE			

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

GPC Batch: _____ GPC RUN Date: _____ GPC CAL Date: _____ GPC INST#: _____ Filter Brand: _____ Filter Lot#: _____

Florisil Batch: _____ Florisil RUN Date: _____ Water Florisil Lot #: _____

Acid Wash Batch: _____ Acid Wash By: _____ Acid Wash Date: _____

Derivatization By: _____ Derivatization Date: _____

Date/Time Started: _____

Date/Time Stopped: _____

Manuf. and lot # of reagents/solvents used: _____

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Note: This page subject to change without notice.

SOP 2.2.4.10

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 192 SOP Section #: 2.2.4.10 Revision #: 9

SOP Title: GC/ECD Analysis of Organo Chlorine Pesticides in Water and Soil Extracts by SW-846 Method 8081B

Effective date: (QA fills in) 4/11/11

- Procedure prepared by: Teresa Ament Jennings Date: 4/11/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 4/11/11
- Reason for change: Removal of Method 8081A
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Edition Update IV Method 8081B, 2/07; Dept. of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 192: GC/ECD Analysis of Organochlorine Pesticides in Water and Soil Extracts by SW-846 Method 8081B

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Instrument Procedure 192: GC/ECD Analysis of Organochlorine Pesticides in Water and Soil Extracts by SW-846 Method 8081B

1.0 Scope and Application

The Standard Operation Procedure describes the procedures used to determine the concentration of various organochlorine pesticides in extracts of solid and liquid sample matrices. Open-tubular, capillary columns are employed with electron capture detectors (ECD). Compound identification is based on dual column confirmation, with the second column being of a dissimilar nature. See Attachment 1 for a list of the analytes determined using this SOP and their reporting limits.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

2.0 Summary of Method

2.1 A measured volume of sample (1 L for liquids, 2 to 30 grams for solids) is extracted using a separatory funnel for water and either an automated Soxhlet or Ultrasonic Extraction method, for soils. A variety of cleanup steps may be used, including Gel Permeation Chromatography (GPC). After cleanup, the extract is analyzed by injecting 2-µL (1-µL per column) into a GC with dual wide-bore, fused silica, capillary columns with dual electron capture detectors (GC/ECD). The instrument hardware is configured to allow two analytical columns joined to a single injection port for simultaneous dual column analysis.

Samples are prepared following the procedures in Sample Preparation Procedure -069," Sample Preparation for Pesticides/PCBs in Water by SW 846" or Sample Preparation Procedure –733, "Low Level Preparation for Analysis of Pesticides/PCBs in Soil/Sediment/Sludge by SW-846."

3.0 Definitions

3.1 Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte **(40 CFR, Part 136, Appendix B*)***. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL $<$ LOD $\leq$ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – µg/L and µg/Kg

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and South Carolina Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single extraction batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 Extraction Batch – a group of to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.9 SC DHEC – South Carolina Department of Health and Environmental Control

3.10 DoD-QSM – Department of Defense Quality Systems Manual

3.11 Ohio VAP – Ohio Environmental Protection Agency Voluntary Action Program

3.12 Marginal Exceedance – Value outside the LCS control limit but within the marginal exceedance limits (4 standard deviations around the mean). This outside boundary prevents a grossly out-of-control LCS from passing. (See Attachment #4)

3.13 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Interference in this method can be grouped into three broad categories: contaminated solvents, reagents, or glassware; contaminated GC carrier gas, parts, column surfaces, detector surfaces; and the presence of co-eluting compounds in the sample matrix that also are detected by the ECD. Specific cleanups may be necessary for samples, depending on the compounds of interest.

4.2 Interferences by phthalate esters introduced during the sample preparation procedures can be a major problem including, but not limited to: common flexible plastics found in certain gloves and other objects containing varying amounts of phthalates that may be introduced during lab operations; and cross-contamination of clean glassware routinely occurring, when plastics are handled during extraction steps, especially when solvent wetted surfaces are handled. These interferences can be minimized by cleanup of solvents, reagents, and glassware.

4.3 Glassware must be scrupulously cleaned as soon as possible after use by rinsing with the last solvent used. This should be followed by detergent washing with hot water, and rinses with tap water and organic-free reagent water. Drain the glassware and dry in an oven at 130° C for several hours or rinse with methanol and drain.

4.4 The presence of elemental sulfur will result in broad peaks that interfere with early eluting pesticides, and can be removed by cleaning the extract with TBA clean up.

4.5 Waxes, lipids, and other high molecular weight compounds are also removed using GPC.

4.6 Technical **Chlordane** and Toxaphene are multi-component analytes. When samples contain more than one multi-component analyte, a more experienced analyst may be required to process/assess the data. Similar conditions exist when multi-component analytes have been subjected to environmental degradation.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Gas chromatograph: Agilent Technologies 6890N and Thermoquest Trace GC 2000 gas chromatographs suitable for on-column and splitless injection with all required accessories, including syringes, analytical columns, gases, ECDs, and or data system.

6.2 The following wide-bore columns or equivalent are used in the analysis:

6.2.1 Column 1 – 30 m x 0.32 mm ID fused silica capillary column bonded with 35% phenyl methylpolysiloxane (clpest), 0.50 µm film thickness.

6.2.2 Column 2 – 30 m x 0.32 mm ID fused silica capillary column chemically bonded with 50% phenyl methylpolysiloxane (clpest2), 0.42 µm film thickness.

6.2.3 Wide-bore columns are installed in a 1/4 inch injectors, with deactivated liners designed specifically for use with mega-bore columns.

6.3 Restek Y-shaped fused silica connector.

6.4 Data system: Standards and samples are processed using the EZ Chrom® Elite data acquisition system, Target® Quick Forms report generation software from ThruPut Systems, and Element **DataSystem**® LIMS **by Promium**, plus revisions.

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook (22F) or Promium Element LIMS.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2-6 ^{o}C in the reach-in cold storage units in the preparation and organic standards laboratories when not in use. All stock standard**s from the manufacturer** must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if QC indicates a problem.

Note**:** All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent**, unless otherwise denoted**. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this SOP. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Reagent or pesticide grade chemicals must be used for all tests.

7.3 Working Standards

7.3.1 Commercially available standards are purchased from Restek and are received with manufacturer's certificates of analysis. The certificate documentation is retained for reference purposes. Working standards are prepared from the commercial standards in the organic standards preparation lab.

Note: The concentration and composition of standards used during the sample preparation and cleanup are provided in the sample preparation SOPs. Standards must equilibrate to room temperature before use.

7.3.**2** Performance Evaluation Mixture (PEM)

7.3.2.1 Prepare in hexane every six months, or sooner, if the solution has degraded or concentrated.

Compounds	Concentration (ng/mL)
4,4′-DDT	3
Endrin	3
Tetrachloro-m-xylene	20

Compounds	Concentration (ng/mL)
Decachlorobiphenyl	40

7.3.**3** Individual Standard Mixture C (INDC)

7.3.3.1 Prepare in hexane every six months or sooner, if the solution has degraded or concentrated.

7.3.3.2 The low calibration standard concentration corresponds to the RL, the midpoint concentration must be 4x the low point concentration and the high point concentration must be at least 16x the low point concentration. The standard is prepared every six months, or sooner if the solutions have degraded or concentrated.

INDC (ηg/mL)					
Compound	**CS1**	**CS2**	**CS3**	**CS4**	**CS5**
alpha-BHC	5.0	10	20	40	80
Heptachlor	5.0	10	20	40	80
gamma-BHC	5.0	10	20	40	80
Endosulfan I	5.0	10	20	40	80
Dieldrin	10	20	40	80	160
Endrin	10	20	40	80	160
4,4'-DDD	10	20	40	80	160
4,4'-DDT	10	20	40	80	160
Methoxychlor	50	100	200	400	800
beta-BHC	5.0	10	20	40	80
delta-BHC	5.0	10	20	40	80
Aldrin	5.0	10	20	40	80
Heptachlor Epoxide	5.0	10	20	40	80
alpha-Chlordane	5.0	10	20	40	80
gamma-Chlordane	5.0	10	20	40	80
4,4'-DDE	10	20	40	80	160
Endosulfan Sulfate	10	20	40	80	160
Endrin Aldehyde	10	20	40	80	160
Endrin ketone	10	20	40	80	160
Endosulfan II	10	20	40	80	160
Tetrachloro-m-xylene	5.0	10	20	40	80
Decachlorobiphenyl	10	20	40	80	160

NOTE: Only the exo-epoxy isomer (isomer B) of Heptachlor Epoxide is used as an analytical standard.

INDC – Individual Standard Mixture C
CS – Calibration Standard

7.3.5 Toxaphene and Technical Chlordane

Multicomponent Analytes (ηg/mL)

Analyte	Low	Med1	Med2	Med3	High
Toxaphene	500	1000	2000	4000	8000
Tech. Chlordane	160	200	400	800	1600
Tetrachloro-m-xylene	5	10	20	40	80
Decachlorobi-phenyl	10	20	40	80	160

7.3.6 Separate calibration standards are used for Toxaphene and Technical **Chlordane** (if required). Alpha and gamma chlordane are included in the single component standard solutions.

7.4 A standard prepared from a source separate from that used to prepare the calibration standard is analyzed after each initial calibration for verification. This standard is referred to as the initial calibration verification (ICV) standard.

7.4.1 An ICV must be analyzed for every target compound within a five-point calibration curve.

7.5 Surrogate standards are added to all samples, method blanks, matrix spikes, laboratory control samples, and calibration standards.

7.5.1 Decachlorobiphenyl (DCB) at 1.2 µg/mL and Tetrachloro-m-xylene (TCX) at 0.6 µg/mL are the surrogates used. Corrective action is taken when both surrogates are recovered outside of acceptance criteria on both columns.

7.5.2 For surrogate standard preparation details, refer to Sample Preparation Procedure -069, "Sample Preparation for Pesticides/PCBs in Water by SW-846".

7.6 All standard solutions are stored separately from samples at 2-6 ^{o}C in Teflon®-sealed containers in the dark. All stock standard solutions must be replaced after one year or sooner if routine QC indicates a problem. All other standard solutions must be replaced after six months or sooner if QC indicates a problem.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be analyzed within 40 days of extraction.

8.3 All sample extracts are stored in the refrigerator in the laboratory at 2 – 6 ^{o}C prior to analysis. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Breakdown

9.1.1 The breakdown standard (PEM) must be analyzed initially with the calibration curve and at least every 12 hours within the analytical sequence.

9.1.2 Degradation of Endrin and DDT is indicated by the presence of 4,4'-DDE, 4,4'-DDD, Endrin aldehyde, or Endrin ketone and is caused by the injector port becoming contaminated with high boiling residue from sample injection.

9.1.2 Degradation of either Endrin or 4,4'-DDT must not exceed 15%. (See **Sections** 12.13 and 12.14 for calculations)

9.1.2.1 If either the degradation of either Endrin or 4,4'-DDT exceed 15%, replace the liner and clean the injection port.

9.2 Surrogates

9.2.1 Statistical control limits for surrogates are shown in the following table. Surrogate recoveries must be met for all samples, blanks, laboratory control samples, matrix spikes, and matrix spike duplicates.

Surrogate	Aqueous % Recovery	Soil % Recovery
Decachlorobiphenyl	43-144	43-144
Tetrachloro-m-xylene	43-135	43-135

9.2.1.1 The surrogate control limits required by the DoD-QSM are presented in the following table:

Surrogate	Aqueous % Recovery	Soil % Recovery
Decachlorobiphenyl	30-135	55-130
Tetrachloro-m-xylene	25-140	70-125

9.2.1.2 Field and QC samples reported with failing surrogates are qualified in the narrative, referencing the applicable DoD-QSM "Q" data flag.

9.2.2 If both surrogates are outside the control range on both columns, the sample must be re-extracted and re-analyzed unless interfering peaks are the reason for the failure. Carefully examine all chromatograms to make certain the out of control surrogate recoveries are not caused by interference or a partial injection before re-extracting the sample. **Only one surrogate out of the four is required to pass.**

9.2.2.1 Partial injections should be re-injected and can be diagnosed by comparing the solvent peak of the sample to the solvent peak of the standard. They should be similar in size.

9.3 Method Blanks (B**LK**)

9.3.1 Method blanks are prepared and analyzed with each extraction batch of up to 20 samples.

9.3.2 No target analyte may be present above the reporting limit and all surrogate criteria stated above must be met.

9.3.2.1 The DoD-QSM requires that no target analyte be present in the method blank > ½ the reporting limit.

9.3.3 If the method blank exceeds acceptance criteria, the source of the problem(s) must be investigated and appropriate corrective action taken. All samples processed with a method blank that does not meet acceptance criteria must be re-extracted and reanalyzed.

9.3.4 Analyte concentration reported for all field and quality control samples that do not meet surrogate acceptance criteria must be qualified as estimates in the SDG narrative. Refer to the DOD-QSM "J" and "Q" flags.

9.4 Laboratory Control Sample (LCS)

9.4.1 A LCS is reagent water or furnaced Ottawa sand or sodium sulfate for solid matrices fortified with the target analyte compounds and surrogates.

9.4.2 The LCS is prepared with each extraction batch of up to 20 samples.

9.4.3 The LCS must meet the control limits of 50 to 150% recovery for the analytes listed in Attachment 4. SC DHEC requires control limits of 70 – 130% recovery. If the client requests Toxaphene or Technical **C**hlordane spike, the acceptance criteria are 50-150% recovery, except SC DHEC which requires 70 – 130% recovery for all analytes.

9.4.3.1 An LCS, analyzed in compliance with the DoD-QSM, must be spiked with all of the analytes of interest in the project. The LCS must meet the control limits presented in Attachment 4, with the allowance that one analyte may fail within the marginal exceedance limit.

9.4.3.2 If the LCS does not meet the acceptance criteria, re-extract and reanalyze the associated samples (if sufficient sample volume is available).

9.4.3.2.1 If corrective actions fail, identify the specific analytes in the associated samples in the SDG narrative.

9.4.3.2.2 To meet the requirements of the DoD-QSM, refer to the "Q" flag.

9.4.3.3 SC DHEC does not accept the use of marginal exceedances, all target analytes must be spiked and must pass all acceptance criteria.

9.5 Matrix Spike/Matrix Spike Duplicate **(MS/MSD)**

9.5.1 A matrix spike and matrix spike duplicate must be performed for each batch of up to 20 samples.

9.5.2 The percent recovery must be within the control limits of 50 to 150. The % RPD of matrix spike duplicates must be ≤ 40. If the client requests Toxaphene or Technical **Chlordane** spike, the acceptance criteria are the same.

9.5.2.1 The objective for the MS/MSD test is for the majority of the spiked compounds to meet recovery and RPD criteria. However, the recoverability of spiked analytes in environmental samples is very much influenced by the particular matrix and, therefore, these objectives may not be able to be met. When recoveries do not meet the acceptance criteria or interference preclude proper assessment of the data, results of a LCS are evaluated to verify that the analytical systems are under control.

9.5.2.2 SC DEHC requires that all target analytes be spiked.

9.5.2.3 SC DEHC requires limits of 70 – 130% recovery for all analytes.

9.5.2.4 If the original sample used for the MS/MSD test does not meet surrogate acceptance criteria, it should be reanalyzed or re-extracted/reanalyzed if the MS/MSD surrogate recoveries are within limits. If the original sample and the MS/MSD test yield the same unacceptable surrogate recoveries, then the sample does not require re-extraction/ reanalysis since matrix interferences is confirmed.

9.6 Duplicate matrix spikes analyzed in compliance with the DoD-QSM must meet the control limits presented for the LCS in Attachment 4. The RPD between the duplicate matrix spikes must be ≤ 30%.

9.6.**1** Results for the specific analytes in the original sample associated with failing matrix spike must be identified in the narrative as estimated values. Refer to the DoD-QSM "J" flag.

9.7 Instrument Maintenance and Trouble Shooting

9.7.1 When linearity is difficult to achieve, verify that the appropriate length of column is inserted in the detector. Examine all column ends and determine if the ends of the columns need to be trimmed.

9.7.2 When the instrument blanks fail, examine the chromatography to determine if there is contamination in the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the contamination is such that baking for 1 hour does not improve the baseline, it may be necessary to change the liner.

9.7.3 When CCV standards fail recovery low, verify that the correct peaks are being named. Also verify that the syringe is not plugged and change the septa. If the CCV standard fails again, examine the chromatography to determine if there is a problem with the baseline that is causing the failure. If so, bake the column for 1 hour or less and run 1-2 hexane blanks to see if this can be corrected. Also, if the CCV standard is failing on only one column, the y-splitter may be plugged. If the calibration verification continues to fail, the instrument will need a new calibration curve.

9.7.4 When CCV standards fail recovery high, verify that the correct peaks are being named. Examine the chromatography to determine if there is contamination on the column that is causing the failure. If so, bake the column for 1 hour or less and run solvents to see if this can be corrected. If the CCV standard fails again, the instrument will need a new calibration curve.

9.7.5 When the CCV standard fails due to drift, change the septum and verify that all the column fittings are secure. Also, determine if there is contamination on the column that is causing the failure. If so, bake the column for 1 hour or less to see if this can be corrected. If the CCV standard still fails, it is permissible to update retention times once per 24 hours period. Record in the instrument run log when the retention times are updated.

9.7.6 All preventive and routine maintenance as mentioned above is recorded in the instrument run log (Attachment 2). Major maintenance is recorded in the maintenance log (Attachment 3).

9.7.7 If the dual capillary columns used in this method are connected by press-fit Y-shaped glass splitters, the splitter should be replaced along with the insert (uniliner) when DDT and/or Endrin degradation is a problem or when CCV standards fail. Along with this, remove the first few inches of the injector port side of the column and pre-column. If this fails to correct the degradation problems, it may be necessary to deactivate the injector port or replace the columns.

9.7.8 Other corrective actions include removal of 0.5 meters of the detector end of the columns when linearity is difficult to achieve.

9.7.9 To silanize the injector when DDT and Endrin degradation is a problem, the following steps should be taken. Remove the analytical columns after cooling the GC oven. Remove the glass insert (uniliner). Lower the injector port temperature to room temperature. Remove any foreign materials observed while inspecting the injector port. Prepare a solution of deactivating agent (Sylon-CT) following the manufacturer's instructions. Place a beaker under the injector port inside the oven, and rinse the inside of the injector port with acetone and then toluene, catching the rinsate in the beaker. Coat the inside of the injector port with the solution thoroughly and allow it to dry. Finally, rinse the injector with toluene, methanol, acetone, and then hexane, in that order. Reassemble the injector and replace the columns.

9.7.10 Because of the relatively low concentration of pesticide standards injected on the GC/ECD, column adsorption may be a problem if the GC has been unused for several days. Deactivation (priming) can be achieved by injecting a standard at least 20 times more concentrated than the mid-level standard before beginning the initial calibration or continuing calibration verification. Run an instrument blank after system priming because analytes may carry over.

9.8 Contingency

9.8.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.8.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.8.3 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization – **all calibration criteria apply to both columns**

10.1 Initial Calibration

10.1.1 The initial calibration consists of a five point curve. The percent Relative Standard Deviation (% RSD) of the calibration factors for the initial calibration must be less than or equal to 20% for all analytes.

10.1.1.**1** Least squares linear regression may be used when the RSDs exceed 20%. When this option is used, the line must not be forced through the origin. The correlation coefficient for the linear fit must be ≥ 0.990 (≥ 0.995 to meet the requirements of the DoD-QSM).

10.2 Initial Calibration Verification

10.2.1 The initial calibration verification (ICV) must be analyzed after each new initial or additional calibration for every target compound within a five-point calibration.

10.2.2 Acceptance criteria for the ICV is ± 20% of the true value.

10.**3** Continuing Calibration **Verification (CCV)**

10.3.1 The calibration factors in all continuing calibration verification (CCV) standards must be within ± 20% difference (% D) when compared to the mean calibration factors from the initial calibration on both analytical columns.

10.3.2 If the % D exceeds ± 20%, then corrective action must be taken and new initial calibration must be analyzed.

10.3.3 If any of the continuing calibration components fall outside their retention time windows, which can be updated every 24 hours as needed, the system is out of control and corrective action must be taken to correct the problem. If, after re-injection of the standard following corrective action, the retention times are still outside the windows, a new initial calibration must be analyzed.

10.3.4 All samples must be bracketed by passing CCVs.

10.3.4.1 If a "closing" standard, which is injected after a group of samples, contains target analyte responses that are 20% D for 8081B different, when compared to the mean calibration factor from the initial calibration, and the analyte was not detected in that group of samples, the samples do not require re-injection. However, if the standard is more than 20% D for 8081B below the mean calibration factor from the initial calibration, then re-injection is required.

10.4 Toxaphene

10.4.1 Unless specified for a particular project/program, a single calibration standard of Toxaphene is analyzed to provide pattern recognition information. The mid-point standard (level 4) is used. Additionally, a 5-point calibration can be performed for Toxaphene detected in samples.

10.5 PCBs

When Method 8082A for PCBs is being used in conjunction with Method 8081B, a five-point calibration has to be performed for Aroclor-1660 followed by individual mid-level standards for the remaining Aroclors. See Instrument Procedure 194, "**GC/ECD Analysis of Polychlorinated Biphenyls (PCBs) as Aroclors in Water and Soil Extracts by SW-846 Method 8082A**" for standards and concentrations.

10.5.1 When Method 8082**A** is employed for PCBs a verification standard of AR1660 must be analyzed **at the frequency of the INDC CCV**. The Aroclor standard can be injected after the **INDC** mix.

10.**6** For **continuing** calibration verification, all target analytes must be injected (INDC) with the exception of Toxaphene and Technical Chlordane.

10.**7** If Toxaphene and/or Technical Chlordane are detected in any samples, the mid-concentration level of those standards must be injected. Samples may be required to be re-injected against those mid-concentration standards.

10.**8** The Endrin and DDT breakdown (PEM) standard is included with each **CCV**. PEM is analyzed after the INDC standard.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The analyst must complete the instrument run log. (See Attachment 2) Condition codes are used to document failed and qualified injections. (See Attachment 5)

11.1 Samples are first extracted using Sample Preparation Procedure –069, "Sample Preparation for Pesticides/PCBs in Water by SW-846" or Sample Preparation Procedure –733, "Sample Preparation for Pesticides/PCBs in Soil/Sediment/Sludge by SW-846."

11.2 GC Conditions

11.2.1 Dual Column Analysis

11.2.1.1 The dual column/dual detector approach involves the use of two 30 m x 0.**32** mm ID fused silica open-tubular columns of different polarities, thus having different selectivities towards the target compounds. The columns are connected to a "Y-splitter" and ECD detectors.

11.2.1.2 Column 1:

- Type: clpest
- Dimensions: 30 m x 0.32 mm ID
- Film Thickness: 0.50 µm

11.2.1.3 Column 2

- Type: clpest2
- Dimensions: 30 m x 0.32 mm ID
- Film Thickness: 0.42 µm

11.2.1.4 Carrier gas

- flow rate – 6 mL/minute, helium

11.2.1.5 Makeup gas

- flow rate – 20 mL/minute, argon/methane

11.2.1.6 Temperatures

- Temperature program – 150 °C (0.5 minute hold) to 275 °C (10 minute hold) at 7 degrees/minute
- Injector temperature – 200 °C
- Detector temperature – 300 °C

11.2.1.7 Injection volume – **2** µL (**1** µL per column)

11.2.1.8 Solvent – Hexane

11.2.1.9 Detector – Dual ECD's

11.2.1.10 Range – 10

11.3 GC analysis

11.3.1 The GC is set up according to the conditions above. An initial oven temperature at or below 150 ^{o}C is required to resolve the BHC's. A final temperature of 240 – 275 ^{o}C is necessary to elute Decachlorobiphenyl.

11.3.2 The initial calibration is performed according to the conditions discussed above and criteria must be met for both columns.

11.3.3 The calibration is verified, with an independent second source ICV standard after each multipoint.

11.3.4 **A CCV standard is run during each 12 hour shift (after every 10 samples for DoD-QSM requirements) using a mid-level calibration standard. Within the 12-hour period, and based on current GC run conditions, approximately 10 samples will be analyzed. Since an**

analytical sequence may continue as long as instrumental QC criteria are met, the end of one 12-hour sequence is considered to be the beginning of the next 12-hour sequence. The calibration factor must meet acceptance criteria when compared to the initial calibration curve. When this criterion is exceeded, corrective action is required as described in Section 9.7 and may include re-injection of the calibration verification standard or analysis of a new initial calibration.

11.3.5 Each sample analysis must be bracketed with acceptable initial or continuing calibration verification standards.

11.4 Retention time windows

11.4.1 Retention time windows are established for each analyte. The width of the retention time windows should be based on actual retention times of standards, assessed over 72 hours. The windows are defined as ± three times the standard deviation of the absolute retention times of each standard. Analyst experience is critical in the interpretations of chromatograms.

11.4.2 Absolute retention time windows are established for each analyte, including five component peaks for multi-component analytes. For each analyte, the absolute retention time from each standard injected at the beginning of a 24-hour period is used as the midpoint of the window ± three times the standard deviation as determined from above.

11.5 Analyte Identification

If any of the surrogates or spike compounds are missing or failing, check the peak integration in "Target Review". Changes can be made in Target Review and manual peak integration chromatograms can be generated. Any compound for which manual peak integration has been performed will have an "M" flag displayed on the quantitation report. All manual integration must follow the procedures documented in SOP 13.18, "Manual Chromatographic Peak Integration Procedures."

Analyst's initials and the date must appear on each quantitation report page containing an "M" flag. For multiple "M" flags appearing on a quantitation report page, a bracket encompassing the flags can be used, with a single entry of the initials and date. The department supervisor or a representative

of the QA department must approve of all manual integrations performed on all initial and continuing calibration standards. This is documented by initialing and dating each page of the quantitation report of the raw data containing such manual integrations. See Attachment 6 for definitions of manual integration codes

11.5.1 Tentative identification of an analyte occurs when a peak from a sample extract falls within the estimated retention time window on both analytical columns. The **lower** concentration of the two columns is reported provided there is no interference from overlapping peaks.

11.5.2 Identification of multi-component analytes is based on the characteristic pattern and retention times of the indicator peaks.

11.6 Analyte quantitation

11.6.1 The external calibration procedure is used and establishes mean calibration factors for each analyte from the initial calibration. Peaks falling within an analyte's retention time window are used to calculate the quantity present. The concentration reported is the higher of the two values obtained from the dual column analysis. If one result is > 40% different, verify that there are not overlapping peaks causing this to occur. Also examine the baseline to verify that the baseline parameters are set properly. Flag the data with a "P" and document results in the narrative. Refer to the "J" flag for results reported in compliance with the DoD-QSM.

11.6.2 If the response of an analyte exceeds the on column amount of the high level standard, dilute and re-analyze. This can be done by comparing a component peak area to the corresponding analyte in the high level standard. Within the Target data system the "**E**" flag is used on the quantitation report to indicate that an analyte is outside the upper initial calibration range. **This criteria applies independently to both columns, even if the reporting column is not above the calibration range.**

11.6.3 Quantitation of multi-component analytes

11.6.3.1 Toxaphene and **Technical Chlordane** are quantitated using response factors of five major peaks (minimum of three required) of the sample pattern compared to the response factors of five peaks in the standard.

11.6.3.2 Quantitation is based on the area under the characteristic Toxaphene and Technical Chlordane peaks as compared to the area under the corresponding calibration peaks at the same retention times using the external calibration method.

11.6.3.3 Quantify Aroclors analyzed together with pesticides using the approach stated in sections 11.6.3.1 and 11.6.3.2.

11.6.4 A normal initial calibration sequence only contains a mid-concentration level of Toxaphene and Technical Chlordane. On a project-specific basis, five levels of Technical Chlordane and Toxaphene may be required.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\% R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked} \times 100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} \times 100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ value}}{\overline{Reference\ value}} \times 100$$

12.7 Calibration factors for GC analysis are calculated by dividing the total peak area for each chosen compound in the standard by the total mass injected (in nanograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.8 Linear Calibration using Least Squares Regression

General linear equation: $y = ax + b$

where:
y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Mass of the analyte in the calibration standard aliquot injected
b = y-intercept

Linear Regression by Least Squares:

$$y_1' = ax_i + b$$

Where: b = intercept
x_i = Mass of the analyte in the ith calibration standard aliquot injected
$y_1^{'}$ = calculated response for the ith calibration standard

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{N}(y_i - y_1^{'})^2$$

12.9 Correlation Coefficient r:

$$r = \frac{N\left[\sum_{i=1}^{N}(x_i y_i)\right] - \left[\sum_{i=1}^{N}(x_i)\right]\left[\sum_{i=1}^{N}(y_i)\right]}{\sqrt{N\left[\sum_{i=1}^{N}(x_i^2)\right] - \left[\sum_{i=1}^{N}(x_i)\right]^2} * \sqrt{N\left[\sum_{i=1}^{N}(y_i^2)\right] - \left[\sum_{i=1}^{N}(y_i)\right]^2}}$$

Where: r = Correlation Coefficient
N = number of data points (equals 5 in a 5 point curve)
y = response
i = index variable (first data point i = 1)
x = Mass of the analyte in the calibration standard aliquot injected

12.10 Calculation of % Difference (% D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \ x\ 100$$

12.11 Calibration factors for GC analysis are calculated by dividing the total peak area for each chosen component in the standard by the total mass injected (in nanograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.12 Concentration

12.12.1 Concentration of aqueous samples for GC analysis

$$\mu g / L = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Vs)}$$

where: Ax = area response for the analyte
Vt = volume of the concentrated extract (**µL**)
Df = dilution factor. If no dilution, Df = 1.0
$\overline{CF}$ = mean calibration factor from the initial calibration
Vo = volume of water sample extracted (mL)
Vi = volume of extract injected (**µL**)

12.12.2 Concentration of soil samples (dry weight basis) for GC analysis

$$\mu g / kg = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Ws)(D)}$$

where: Ws = weight of sample extracted, in grams

$$D \text{ (dry weight)} = \frac{100 - \%\text{ moisture}}{100}$$

Ax, Vt, Df, $\overline{CF}$, Vi have the same definitions as for water.

12.12.3 Concentration of aqueous samples for GC analysis using linear regression:

$$ug / L = \frac{(a(Ax) + b)(Vt)(Df)}{(Vi)(Vo)}$$

where: Ax = area response for the analyte
Vt = volume of the concentrated extract (**µL**)
Df = dilution factor. If no dilution, Df = 1.0
a = slope
b = y-intercept
Vo = volume of water sample extracted (**mL**)
Vi = volume of extract injected (**µL**)

12.12.4 Concentration of soil samples for GC analysis using linear regression:

$$ug/kg = \frac{(a(Ax)+b)(Vt)(Df)}{(Vi)(Ws)(D)}$$

where: Ws = weight of sample extracted, in grams
D (dry weight)= 100 - % moisture/100
Ax = area response for the analyte
Vt = volume of the concentrated extract (**µL**)
Df = dilution factor. If no dilution, Df = 1.0
a = slope
b = y-intercept
Vo = volume of water sample extracted (**mL**)
Vi = volume of extract injected (**µL**)

12.13 Endrin Breakdown:

$$\%\text{ breakdown of Endrin } = \frac{\text{sum of degradation peak areas (Endrin aldehyde + Endrin ketone)}}{\text{sum of all peak areas (Endrin + Endrin aldehyde + Endrin ketone)}} \times 100$$

12.14 DDT Breakdown:

$$\%\text{ breakdown of DDT } = \frac{\text{sum of degradation peak areas (DDD + DDE)}}{\text{sum of all peak areas (DDT + DDD + DDE)}} \times 100$$

12.15 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. **This criteria applies independently to both columns, even if the reporting column is not above the calibration range.** Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x **(1 mL in 10 mL)**."

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.2 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.4 Quality Control SOP 13.4, "Numerical Data Reduction"

16.5 NELAC Standards, effective July 2003, plus revisions

16.6 EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.7 CompuChem Quality Manual, Revision 13, November 17, 2009, plus revisions

16.8 Sample Control SOP 4.6, "Storing Samples"

16.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **10**/**25**/**10**

16.10 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update IV, Method 8081B, February 2007

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – Analyte List and Reporting Limits

17.2 Attachment 2 – Example of a GC Extractables Run Log

17.3 Attachment 3 – Example of a GC Extractables Instrument Maintenance Log

17.4 Attachment 4 – LCS Control Limits Required by the DoD-QSM and Marginal Exceedences

17.5 Attachment 5 – Condition Codes

17.6 Attachment 6 – Manual Integration Codes

Attachment 1

		Reporting Limits	
Compound Name	**CAS Registry No.**	**µg/L**	**µg/Kg**
Aldrin	309-00-2	0.025	0.83
alpha-BHC	319-84-6	0.025	0.83
beta-BHC	319-85-7	0.025	0.83
delta-BHC	319-86-8	0.025	0.83
gamma-BHC (Lindane)	58-89-9	0.025	0.83
alpha-Chlordane	5103-71-9	0.025	0.83
gamma-Chlordane	5103-74-2	0.025	0.83
4,4'-DDD	72-54-8	0.050	1.67
4,4'-DDE	72-55-9	0.050	1.67
4,4'-DDT	50-29-3	0.050	1.67
Dieldrin	60-57-1	0.050	1.67
Endosulfan I	959-98-8	0.025	0.83
Endosulfan II	33213-65-9	0.050	1.67
Endosulfan sulfate	1031-07-8	0.050	1.67
Endrin	72-20-8	0.050	1.67
Endrin aldehyde	7421-93-4	0.050	1.67
Endrin ketone	53494-70-5	0.050	1.67
Heptachlor	76-44-8	0.025	0.83
Heptachlor epoxide	1024-57-3	0.025	0.83
Methoxychlor	72-43-5	0.250	16.7
Technical **Chlordane**	12789-03-06	0.8	26.7
Toxaphene	8001-35-2	2.5	83.3

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Attachment 2

COMPUCHEM a Division of Liberty Analytical Corporation Instrument ID: 86 File Path: _____ DATE ___/___/___
GC EXTRACTABLES RUN LOG Amt. Inj. 2 µL (1 µL per column)
LOGBOOK 4N(5) 23 Method: 8081B 8082A 8151A SOM01.2 (Circle one) Other _____

☐Changed septum ☐Changed liner ☐Baked column ☐Trimmed column ☐ Other: _____

	REJECTED	FILE NUMBER	REPORTED	DATE	COMPUCHEM #	CASE/SDG#	STANDARD ID	CHEMIST	COMMENTS
1				/ /					
2				/ /					
3				/ /					
4				/ /					
5				/ /					
6				/ /					
7				/ /					
8				/ /					
9				/ /					
10				/ /					
11				/ /					
12				/ /					
13				/ /					
14				/ /					
15				/ /					
16				/ /					
17				/ /					
18				/ /					
19				/ /					
20				/ /					

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Calibration ID: _____ Sequence ID: _____ Hexane Lot No_____

REVIEWED BY: _____ DATE _____

5/19/10: jad

4201

Note: This page subject to change without notice.

Attachment 3

CompuChem a division of Liberty Analytical Corp. Logbook 4 K(4) 1

GC Extractables Instrument Maintenance Log: 80/81

Date: _____ Time: _____

System: _____

Problem: _____

_____

_____

_____

_____

Action Taken: _____

_____

_____

_____

_____

_____

Parts used:

Part Number	Description	Quantity

File Name Indicating Instrument In Control: _____

Performed By: _____ Date: _____

Reviewed By: _____ Date: _____

2/11/00:mlj

Attachment 4

DoD-QSM Aqueous and Solid LCS Control Limits and Marginal Exceedance Limits

Analyte	Aqueous % Recovery	Aqueous Marginal Exceedance	Solid % Recovery	Solid Marginal Exceedance
4,4'-DDD	25-150	10-170	30-135	10-155
4,4'-DDE	35-140	15-160	70-125	60-135
4,4'-DDT	45-140	30-155	45-140	30-155
Aldrin	25-140	10-155	45-140	30-155
alpha-BHC	60-130	50-140	60-125	50-135
alpha-Chlordane	65-125	55-135	65-120	55-130
beta-BHC	65-125	55-135	60-125	50-135
delta-BHC	45-135	30-150	55-130	45-145
Dieldrin	60-130	50-140	65-125	55-135
Endosulfan I	50-110	40-120	15-135	10-155
Endosulfan II	30-130	10-150	35-140	20-160
Endosulfan sulfate	55-135	40-150	60-135	50-145
Endrin	55-135	45-145	60-135	50-145
Endrin aldehyde	55-135	40-150	35-145	20-165
Endrin ketone	75-125	70-135	65-135	55-145
gamma-BHC	25-135	10-155	60-125	50-135
gamma-Chlordane	60-125	50-135	65-125	55-135
Heptachlor	40-130	30-145	50-140	35-155
Heptachlor Epoxide	60-130	50-140	65-130	55-140
Methoxychlor	55-150	40-165	55-145	45-155

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Attachment 5

Code	Explaination
CS	Carry over Suspected from prior analysis
CT	Contamination verified
DI	Requires Dilution
DW	Diluition Wrong
JS	Reinjection same as prior run
LA	Lab Accident (explain in comments)
OT	Other (explain in coments)
OW	Original wrong for QC sample
RP	Reportable prior run
RT	Retention time shift
RU	Repeated Unnecessarily
SF	Spike recoveries failed (high or low)
SI	Spiked inadvertently
SH	Surrogate recoveries high
SL	Surrogate recoveries low
SM	Surrogate or spike missing
IF	Instrument failure
US	Unacceptable Standard (explain in comments)
VR	Verify results
NM	Did not match prior run or duplicate
RN	Reanalyze neat, was run at a dilution
PC	Poor chromatography
SC	Sulfur clean up required
WS	Wrong standard (SOM PCBs)
MI	Misinjection (bad injection)

Code	Explaination
%R	%RSD failed (Ical)
%B	Breakdown failed
%D	%D (CCV)
RT	Retention time shift
RU	Repeated Unnecessarily
IF	Instrument failure
US	Unacceptable Standard (explain in comments)
PC	Poor chromatography
SC	Sulfur clean up required
LA	Lab Accident (explain in comments)
OT	Other (explain in coments)

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Attachment 6

CompuChem

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Notification Regarding Manual Editing/Integration Flags

In some instances, manual adjustments to the software output are necessary to provide accurate data. These manual integrations are performed by the data reviewers, GC/MS operators, or GC/HPLC chemists. An Extracted Ion Current Profile (EICP) or a GC/HPLC chromatographic peak has been provided for the manual integration performed on each compound to demonstrate the accuracy of that process. The manual integrations are flagged on the quantitation report in the far right column beyond the FINAL concentration for GC/MS analysis, and in the "Flags" column for GC/HPLC analysis. The manual editing/integration flags are:

M - **Denotes that a manual integration has been performed for this compound. The manual integration was performed in order to provide the most accurate area count possible for the peak. The most common reasons for performing manual integrations/editing are: the compound was not found by the automatic integration routine, the compound was incorrectly integrated by the automatic integration routine, and the co-eluting compounds were incorrectly integrated by the automatic integration routine.**

H - **Denotes that the data reviewer, GC/MS operator, or GC/HPLC Chemist has chosen an alternate peak within the retention time window from that chosen by the software for that compound. No manual integration is performed in choosing an alternate peak. The software still performs the integration.**

MH - **Denotes that an alternate peak has been chosen within the retention time window from that chosen by the software for that compound and also a manual integration of the chosen peak has been performed. The manual integration was performed in order to provide the most accurate area count possible for the peak.**

L - **Denotes that a data reviewer or GC/MS operator has selected an alternate library search. This is typically done when an additional tentatively identified compound (TIC) has been added to the number of peaks searched. No manual integration is performed in choosing an alternate peak. The software still performs the integration.**

ML - **Denotes that an alternate GC/MS library search has been selected and a manual integration has also been performed. This is typically done when an additional TIC has been added and the TIC peak also required a manual integration.**

These codes will appear in the GC/MS and GC/HPLC raw data.

Revision 8 (01/29/2011)

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SOP 2.4.4.5

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 1404 SOP Section #: 2.4.4.5 Revision #: 3

SOP Title: GC/MS Selected Ion Monitoring (SIM) Semivolatile Analyses of Aqueous and Soil Samples Using SW-846

Effective date: (QA fills in) 05-18-11

- Procedure prepared by: [signature] Date: 4/25/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob McEiere Date: 05-18-2011
- Reason for change: Annual Review and response to LA-B audit
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Ed, Update III, Method 8270C and Update IV Method 8270D 1/08; DoD QSM Version 4.2 10/25/2010, plus revisions

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 1404: GC/MS Selected Ion Monitoring (SIM) Semivolatile Analyses of Aqueous and Soil Samples Using SW-846

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Instrument Procedure 1404: GC/MS Selected Ion Monitoring (SIM) Semivolatile Analyses of Aqueous and Soil Samples Using SW-846

1.0 Scope and Application

This procedure is used for the determination and quantitation of low concentrations of semi-volatile compounds in extracts of aqueous and solid samples. Attachment 1 presents the compounds currently analyzed by these methodologies. Additional compounds may be analyzed per client request.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary

This procedure provides for the gas chromatograph mass spectrometer (GC/MS) analysis of aqueous and solid samples using the SW-846, Update III, Method 8270C SIM and Update IV, Method 8270D SIM. Aqueous samples are extracted prior to analysis using the SW-846 separatory funnel extraction, Method 3510C or continuous liquid-liquid extraction, and concentrated to a final volume of 1.0 mL. Soil samples are extracted by the ultrasonic extraction, Method 3550B/C, and concentrated to a final volume of 1.0 mL. Each extract is injected into a GC equipped with a fused silica capillary column, where it is chromatographically separated from other extractable organic compounds. A mass spectrometer detector operating in the selected ion monitoring (SIM) mode analyzes the eluents. SIM analysis is performed on extracts that were prepared for scan analysis, as well as those prepared for SIM analysis only.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B). A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL

must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) – The DL is the smallest analyte concentration that can be demonstrated to be different from zero or a blank concentration at the 99% level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is smallest concentration of an analyte that must be present in a sample in order to be detected at 99% confidence level. The false negative rate at the LOD is 1%. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of the measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported as qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – µg/L for water and µg/Kg for soil

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix is prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 Extraction Batch – a group of up to 20 field samples of a similar matrix, method specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate prepared together at the same time.

3.9 CCC – Calibration Check Compounds

3.10 SPCC – System Performance Check Compounds

3.11 AMU – atomic mass unit

3.12 FC-43 – Perfluorotributylamine

3.13 DoD QSM Department of Defense Quality Systems Manual

3.14 SC DHEC – South Carolina Department of Health and Environmental Control

3.15 LIMS – Laboratory Information Management System

3.16 For the purposes of this SOP, DFTPP refers to an instrument check solution containing Pentachlorophenol, Benzidine, and 4,4'-DDT.

4.0 Interferences

4.1 This method is applicable to the analysis of aqueous and solid samples containing low total concentrations of extractable organic compounds. If interferences are detected from other sample constituents, the GC conditions may be adjusted to reduce or eliminate the interference. Extract clean-up by gel permeation chromatography (GPC) may be attempted following Sample Preparation Procedure –939, "Gel Permeation Chromatography (GPC) Cleanup of Semivolatile (SV) Soil Sample Extracts by CLP, SW-846".

4.2 The client must be aware that in some cases analysis for low concentrations of semi-volatile compounds will not be possible because of matrix interferences.

5.0 Safety

5.1 The analysts and laboratory management must be aware of the proper techniques used to handle hazardous chemicals and samples.

5.2 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Mass spectrometer

6.1.1 Hewlett Packard 5972A mass selective detector (MSD)

6.1.2 Agilent 5973N mass selective detector (MSD)

6.2 Gas Chromatograph

6.2.1 Hewlett Packard 5890 Series II gas chromatograph (GC) with electronic pressure control (EPC)

6.2.2 Agilent Technologies 6890N series with EPC.

6.3 Analytical Column

6.3.1 Fused silica 30 m (length) Restek RTX-5MS, 0.25 mm (ID), 0.25 µM (film thickness) or RTX-5 Sil ms

6.3.2 Fused silica 30 m (length) Restek RTX-5MS, 0.32 mm (ID), 0.25 µM (film thickness) or RTX-5 Sil ms

6.4 Autosampler

6.4.1 Hewlett Packard 7673 Automated Liquid Sampler

6.4.2 Agilent 7873B Automated Liquid Sampler

6.5 Data System

6.5.1 A computer is interfaced to the mass spectrometer to allow the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

6.5.2 The data processing computer has software that searches any GC/MS data file for ions of a specified mass and plots ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software integrates the abundance of any EICP between specified time or scan number limits.

6.5.2.1 For data processing, the laboratory uses the Hewlett Packard HP 9000 series 735 Unix Workstation.

6.5.2.2 Standards and samples are processed using the EZ Chrom® Elite data acquisition system, Target® Quick Forms report generation software from ThruPut Systems, and Promium Element® LIMS.

6.6 Syringes - 10 µL Hamilton syringe

7.0 <u>Reagents and Standards</u>

All standards are prepared by the Organic Standards Laboratory. Details for the standard preparation are contained in the Standards Preparation Logbook (22F) or Promium Element® LIMS. Standards are stored separately from samples at 2 – 4 ^{o}C in the laboratory when not in use. Stock solutions must be prepared every three to six months or sooner, if comparisons with quality control check samples indicate degradation or concentration of solution compounds.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration. Standards may be purchased as certified neat materials or concentrated solutions. Commercially-prepared stock standard solutions should be obtained whenever possible in order to avoid the hazard of handling neat explosive material. All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type I with regard to resistivity of > 10 megohm-cm (19th and 20th Editions of Standard Methods, Method 1080), which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methylene Chloride

7.3 Standards

7.3.1 Calibration standard solutions are prepared at 0.2, 0.6, 1.5, 2.5, 5.0 µg/mL for the majority of compounds. See Attachment 4 for details on the standard concentrations.

7.3.2 Internal standard solution is prepared at 40 µg/mL.

7.3.3 Standards are prepared every six months or sooner if degradation or evaporation occurs.

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be analyzed within 40 days of extraction.

8.3 Prior to analysis, all extracts must be stored under refrigeration at 2 – 4 ^{o}C in the reach-in storage unit in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Surrogates

9.1.1 Surrogates are added to each sample prior to extraction.

9.1.2 Samples requiring scan analysis prior to SIM analysis have surrogates added at the scan concentration and the recoveries are assessed in the scan analysis.

9.1.3 Samples requiring SIM analysis only have surrogates added at the SIM concentration and recoveries are assessed in the SIM analysis.

9.1.4 The recovery of each surrogate is monitored. Surrogate recovery criteria must be met or the affected sample must be re-extracted and reanalyzed. If surrogate recovery is not met in the method blank or LCS, the entire batch must be re-extracted and re-analyzed. Surrogate recovery limits are presented in the following table for SW-846 analyses.

Compound	Aqueous Acceptance Limits	Soil Acceptance Limits
Nitrobenzene-d_5	34-114	25-136
2-Fluorobiphenyl	55-110	29-131
Terphenyl-d_{14}	53-110	34-149
2, 4, 6-Tribromophenol	25-120	22-143

9.1.5 Surrogate control limits required by the DoD-QSM are listed in the following table.

Compound	Aqueous Acceptance Limits	Soil Acceptance Limits
Nitrobenzene-d_5	40-110	35-100
2-Fluorobiphenyl	50-110	45-105
Terphenyl-d_{14}	50-135	30-125
2, 4, 6-Tribromophenol	40-125	35-125

9.2 Internal Standards

9.2.1 In each method blank, laboratory control sample (LCS), matrix spike, and sample run immediately after the initial calibration, the internal standard area responses must be greater than 50% and less than 200% of the area of the internal standard in the mid level initial calibration standard.

9.2.2 For each method blank, LCS, matrix spike, and sample run after a continuing calibration verification standard, the internal standard area responses must be greater than 50% and less than 200% of the area of the internal standard in the continuing calibration verification standard.

9.2.3 The retention time of each internal standard in each method blank, LCS, matrix spike, and sample must be within ± 0.5 minutes (30 seconds) of its corresponding internal standard in the calibration standard.

9.2.4 If the internal standard criteria are not met, the extract must be re-injected. When the internal standard fails in the same way in the re-injection of a sample, a matrix effect is confirmed.

9.3 Method Blank

9.3.1 A method blank is prepared with each extraction batch of samples. The concentrations of target compounds (see Attachment 1) in the method blank prepared for SIM analysis only must be less than the RL for SW-846 analyses.

9.3.1.1 Blanks that were originally prepared for scan analysis may contain target compounds at concentrations above the RL or CRQL.

9.3.1.2 To meet the requirements of the DoD-QSM, all target analytes must be at concentrations less than half the reporting limit.

9.3.2 Internal standard responses must be within acceptance windows.

9.3.3 If the method blank prepared for SIM analysis fails acceptance criteria for surrogate recovery or contamination, the analytical system is considered to be out of control. The source of the contamination must be investigated and appropriate corrective measures taken and documented before samples are analyzed. All samples processed with a method blank that is

out of control must be re-extracted and reanalyzed, unless the method blank meets acceptance criteria upon re-injection.

9.4 Laboratory Control Sample

9.4.1 A laboratory control sample (LCS) is extracted with each SW-846 batch of samples. An LCS is spiked with a subset of the compounds listed in the Table from Section 9.4.2.1, unless otherwise specified by the project.

9.4.2 The recovery of each spike analyte is monitored.

9.4.2.1 If the LCS fails, all samples analyzed in the batch must be re-extracted and reanalyzed. LCS recovery limits are presented in the following table.

Compound	Aqueous Acceptance Limits	Soil Acceptance Limits
Naphthalene	61-107	36-149
2-Methylnaphthalene	58-100	31-150
1-Methylnaphthalene	60-109	33-134
Acenaphthylene	62-112	35-148
Acenaphthene	65-111	34-147
Fluorene	70-116	34-150
Phenanthrene	71-119	37-144
Anthracene	67-112	38-146
Fluoranthene	63-109	33-150
Pyrene	64-108	35-144
Benzo(a)anthracene	66-114	36-145
Chrysene	67-111	40-140
Benzo(b)fluoranthene	53-148	32-149
Benzo(k)fluoranthene	51-111	30-150
Benzo(a)pyrene	62-129	35-143
Indeno(1,2,3-c,d)pyrene	77-125	25-112
Dibenzo(a,h)anthracene	67-131	34-146
Benzo(g,h,i)perylene	61-119	34-139
4-Nitrophenol	10-150	10-150
Pentachlorophenol	32-150	23-150
Hexachlorobutadiene	29-107	11-137
Hexachlorobenzene	27-101	15-148

9.4.2.2 DoD LCS recovery limits are presented in the following table.

Compound	Aqueous Acceptance Limits	Soil Acceptance Limits
Naphthalene	40-100	40-105
2-Methylnaphthalene	45-105	45-105
1-Methylnaphthalene	N/A	N/A
Acenaphthylene	50-105	45-105
Acenaphthene	45-110	45-110
Fluorene	50-110	50-110
Phenanthrene	50-115	50-110
Anthracene	55-100	55-105
Fluoranthene	55-115	55-115
Pyrene	50-130	45-125
Benzo(a)anthracene	55-110	50-110
Chrysene	55-110	55-110
Benzo(b)fluoranthene	45-120	45-115
Benzo(k)fluoranthene	45-125	45-125
Benzo(a)pyrene	55-110	50-110
Indeno(1,2,3-c,d)pyrene	45-125	40-120
Dibenzo(a,h)anthracene	40-125	40-125
Benzo(g,h,i)perylene	40-125	40-125
4-Nitrophenol	0-125*	15-140
Pentachlorophenol	40-115	25-120
Hexachlorobutadiene	25-105	40-115
Hexachlorobenzene	50-110	45-120

* Batch acceptance should not be evaluated using these limits.

9.5 Matrix Spike/Matrix Spike Duplicate

9.5.1 If requested by the client a matrix spike and matrix spike duplicate (MS/MSD) are extracted and analyzed with a SDG.

9.5.2 The percent recovery limits for spiked compounds are the same as those for the LCS.

9.5.3 The relative percent difference between the MS and the MSD should be less than or equal to 50.

9.5.4 The objective for the MS/MSD test is for the majority of the spiked compounds to meet recovery and RPD criteria. However, the recoverability of spiked analytes in environmental samples is very much influenced by the particular matrix and, therefore, these objectives may

not be able to be met. When recoveries do not meet the acceptance criteria or interference preclude proper assessment of the data, results of a LCS are evaluated to verify that the analytical systems are under control.

If the original sample used for the MS/MSD test does not meet surrogate acceptance criteria, it should be reanalyzed or re-extracted/reanalyzed if the MS/MSD surrogate recoveries are within limits. If the original sample and the MS/MSD test yield the same unacceptable surrogate recoveries, then the sample does not require re-extraction/ reanalysis since matrix interferences is confirmed.

9.6 Contingency

9.6.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.6.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.6.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.6.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 <u>Calibration and Standardization</u>

10.1 Instrument Tuning

10.1.1 Tuning is done for the purpose of demonstrating proper mass axis alignment. The instrument must be tuned to meet the manufacturer's specifications through mass assignment of the three key ions of the tuning compound. The mass drift from the true values of the major ions of FC-43 must be no more than 0.10 AMU. The peak width should be ± 0.2 of 0.50.

10.1.2 A twelve-hour tune window is observed starting from the time of injection of the DFTPP solution. See Sections 10.1.3 and 10.1.4 **for DoD analyses or for the generation of the FC-43 report for non-DoD analyses.**

10.1.3 The DoD-QSM requires evaluation of a solution containing benzidine, pentachlorophenol and 4,4'-DDT to assess the condition of the column and injection port. This is accomplished by injecting the DFTPP solution that contains the three (3) compounds.

10.1.3.1 Benzidine, pentachlorophenol, and 4,4´-DDT are used as evaluation aids to indicate injector port and GC column efficiencies.

10.1.3.2 Excessive 4,4´-DDT breakdown and poor peak shape and response for benzidine and pentachlorophenol indicate maintenance is required for the injector port or GC column.

10.1.4 The following breakdown and tailing factor criteria must be met for the analysis to be acceptable.

10.1.4.1 The % breakdown for DDT into DDE and DDD must not exceed 20%.

10.1.4.2 Acceptance Criteria for the tailing factor for benzidine

10.1.4.2.1 The tailing factor for benzidine must not exceed 2.0 for all DoD analyses.

10.1.4.3 Acceptance Criteria for the tailing factor for pentachlorophenol.

10.1.4.3.1 The tailing factor for pentachlorophenol must not exceed 2.0 for all DoD analyses.

Note: When the breakdown or tailing factor criteria are not met it is indicative that instrument maintenance is necessary for the column and/or the injector port.

10.1.5 After Section 10.1.4 criteria are met, generate a printout demonstrating FC-43 criteria are met.

10.2 Initial Calibration

10.2.1 Prior to sample analysis, an initial calibration curve consisting of **five** levels of standards for SW-846 must be analyzed. The calibration curve consists of the following concentrations: 0.4, 1.2, 3.0, 5.0, and 10.0 nanograms (ηg) on column.

10.2.2 Initial calibration acceptance criteria for SW-846 SIM

10.2.2.1 For SW-846, the %RSD for the RRFs in the initial calibration must be < 15% for 8270C and < 20% for 8270D, in order for the average relative response factor to be used for quantitation. For DoD, the %RSD must be < 15%.

10.2.2.1.1 If the % RSD is > 15% for 8270C and > 20% for 8270D (> 15% for DoD), then an alternate method for quantitation, such as a linear calibration using least squares regression or a non-linear calibration method, such as quadratic fit, may be used. When one of these options is used, the line must not be forced through the origin**,** and the correlation coefficient of the equation must be 0.99 or greater (0.995 or greater for the DoD-QSM) for a valid calibration. If a quadratic equation is used, six levels of standards must be used. These alternate methods of quantitation are available in the ThruPut system.

10.2.2.2 Minimum response factor requirements for the typical analytes of this analysis are given in the following table.

Minimum Response Factor in Initial Calibaration

	8270C	DOD-QSM	8270D
Naphthalene	0.01	0.01	0.7
Hexachlorobutadiene	0.01	0.01	0.01
2-Methylnaphthalene	0.01	0.01	0.4
1-Methylnaphthalene	0.01	0.01	0.01
Acenaphthalene	0.01	0.01	0.9
Acenaphthene	0.01	0.01	0.9
4-Nitrophenol	0.05	0.05	0.01
Fluorene	0.01	0.01	0.9
Hexachlorobenzene	0.01	0.01	0.1
Pentachlorophenol	0.01	0.01	0.05
Phenanthrene	0.01	0.01	0.7
Anthracene	0.01	0.01	0.7
Fluroanthene	0.01	0.01	0.6
Pyrene	0.01	0.01	0.6
Benzo(a)anthracene	0.01	0.01	0.8
Chrysene	0.01	0.01	0.7
Benzo(b)fluroanthene	0.01	0.01	0.7
Benzo(k)fluoranthene	0.01	0.01	0.7
Benzo(a)pyrene	0.01	0.01	0.7
Indeno(1,2,3-cd)pyrene	0.01	0.01	0.5
Dibenzo(a,h)anthracene	0.01	0.01	0.4
Benzo(g,h,i)perylene	0.01	0.01	0.5

Note: The minimum response factor must be met at all calibration levels for Method 8270D.
For 8270C and DOD-QSM the minimum average response for 4-Nitrophenol must be 0.05 or greater if it is being analyzed. The remaining criteria are Compuchem in house criteria.

10.2.3 **Method 8270D cites the EPA CLP Organics SOW as a source of guidance in performing SIM analyses. Based on the acceptance criteria of the current CLP Organics SOW (SOM01.2), up to two**

target compounds and surrogates may fail to meet the minimum RRF criteria, as long as they still meet a minim RRF of 0.010.

10.2.4 If initial calibration criteria are not met, instrument maintenance may be required prior to recalibration.

10.3 Continuing Calibration

10.3.1 Samples may be analyzed immediately after a valid initial calibration if there is sufficient tune time remaining. Otherwise, a continuing calibration verification standard must be run to demonstrate that the instrument is in control.

10.3.2 Before running the continuing calibration verification (CCV) standard, follow the instrument tuning Section 10.1.

10.3.3 The continuing calibration verification standard is the 3.0 ηg level.

10.3.4 Continuing calibration verification acceptance criteria.

10.3.4.1 For SW-846, the percent difference (%D) for the continuing calibration for all compounds must be ≤ 20%.

10.3.4.2 The minimum RRF for all compounds is the same as in the initial calibration**, with up to two compounds allowed out for Method 8270D, as described in Section 10.2.3.**.

11.0 Procedure

11.1 Documentation must follow the requirements in Quality Control SOP 13.6 "Proper Documentation Procedures". Complete the instrument run log (Attachment 3) with the information for the tune sequence.

11.2 Define the retention time (RT) windows needed for SIM analysis, by analyzing a calibration standard in the scan mode first. Switch the MS to SIM mode. Using the compound RTs from the scan analysis, edit the RT windows in the GC/MS SIM analysis method.

11.3 Optimize GC conditions so that chromatographic peaks are as sharp and symmetrical as possible.

11.4 Prior to the analysis of any calibration standards, method blanks, matrix spikes, or samples, the GC/MS system must be tuned by the acquisition of FC-43. This

marks the beginning of the twelve-hour analysis period. Print a hard copy of the FC-43 scan.

11.4.1 For the DoD-QSM, inject a solution containing benzidine, pentachlorophenol, and 4,4'-DDT and evaluate as indicated in Section 10.1.3.

11.5 After the acquisition of FC-43, analyze the initial calibration.

11.5.1 Allow standard solutions to equilibrate to room temperature. Aliquot each calibration standard, 0.2, 0.6, 1.5, 2.5, and 5.0 µg/mL, in an autosampler vial containing a conical insert. The injection volume is 2.0 µL. This will yield a ηg on column amount of 0.4, 1.2, 3.0, 5.0 and 10.0 respectively, for each analyte.

11.5.2 Process and review each standard analysis using the Target3 data system insuring that each surrogate and target compound is present in each standard.

Note: Attachment 1 contains all of the target compounds for which the laboratory analyzes for by SIM. However, only a subset of this list may be analyzed for at one time.

11.5.3 Check that the initial calibration met acceptance criteria by reviewing the initial calibration summary form on the Target3 data system.

11.5.4 If time remains in the twelve-hour period after the initial calibration has been analyzed, samples may be analyzed without the analysis of an continuing calibration standard.

11.5.5 If no samples are analyzed, complete the GC/MS run log. (Attachment 3). "Z" out any unused portion of the run log and have a supervisor or designee review and sign it. Make a photocopy of the run log and attach it to the hard copy data for the tune.

11.6 After the analysis and acceptance of the initial calibration, analyze the continuing calibration.

11.6.1 Process and review the continuing calibration standard using Target3 processing software insuring that each target compound is present in the analysis.

11.6.2 Check that the continuing calibration standard met acceptance criteria, by reviewing the continuing calibration summary form on the Target3 data system.

11.7 After the analysis and acceptance of the initial and continuing calibrations, analyze the method blanks (LCSs, if required), matrix spikes, and samples. Allow all extracts to equilibrate to room temperature prior to analysis.

11.7.1 To prepare a method blank, LCS, matrix spike or sample extract for SW-846 analysis, add 100 µL of the sample extract and 2.5 µL of internal standard solution. All injections must be made using the cold needle injection technique. The injection volume is 2.0 µL and this will yield 2 ηg on column of each of the internal standards.

11.7.2 Place each auto sampler vial on the tray on top of the GC/MS for analysis. Set up the analytical sequence by entering lab identifications for each calibration standard, method blank, matrix spike, and sample in to the ChemStation data system.

11.7.3 Review each blank analysis, ensuring that it meets all surrogate, internal standard and blank contamination criteria.

11.7.4 Review each LCS and matrix spike analysis, ensuring that it meets surrogate, internal standard, and spike recovery criteria.

11.7.5 Review each sample analysis, ensuring that it meets surrogate, internal standard, and dilution criteria.

11.7.6 At the end of the twelve-hour analysis period, complete the instrument run log (Attachment 3). "Z" out any unused portion of the run log and have the supervisor or designee review and sign it. Photocopy the run log and attach it to the hard copy data for the tune and calibration.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ \mathrm{R} = \frac{Amount\ in\ spiked\ sample\ -\ Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{\left(Value\ 1\ +\ Value\ 2\right)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ \ value}}{\overline{Reference\ \ value}} \times 100$$

12.7 Relative Response Factor

$$RRF = \frac{Ax \times C(is)}{A(is) \times Cx}$$

where: Ax = Area of the characteristic ion (EICP) for the compound to be measured
A(is) = Area of the characteristic ion (EICP) for the specific internal standard
C(is) = Concentration of the internal standard (in µg/L)
Cx = Concentration of the compound to be measured

12.8 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

12.7 Concentration

12.7.1 Concentration of aqueous samples by GC/MS analysis

$$\mu g / L = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(\overline{RRF})(Vo)(Vi)}$$

where: Ax = area of the characteristic ion from the EICP for the compound to be measured
Ais = area of the characteristic ion for the EICP for the internal standard
Is = amount of internal standard injected (**η**g)
$\overline{RRF}$ = mean relative response factor from initial calibration standards
Vo = volume of water extracted (mL)
Vi = volume of extract injected (µL)
Vt = volume of the concentrated extract (µL)
Df = dilution factor. If no dilution, Df = 1.0

12.7.2 Concentration of soil samples (dry weight basis) by GC/MS

$$\mu g / kg = \frac{(Ax)(Is)(Vt)(Df)(2.0)}{(Ais)(\overline{RRF})(Vi)(Ws)(D)}$$

where: Ax, Ais, Is, Vt, Vi, RRF, and Df are the same as given for water
2.0 = GPC factor (if used)
Ws = weight of sample extracted, in grams

$$D \text{ (dry weight)} = \frac{100 - \%\text{ moisture}}{100}$$

12.7.3 Concentration of water and soil samples (dry weight basis) by GC/MS using quadratic (second order) fit in Target:

$$y = [n]\left[b + m^1\ (Rsp) + m^2\ (Rsp\text{^}2)\right]$$

where:
b = constant
m^1 = multiplier for the unsquared term
m^2 = multiplier for the squared term
x = area of analyte/area of Internal Standard
n = amount of Internal Standard
y = concentration amount in ηg on column
Rsp = area of analyte/area of Internal Standard

Example:
Area of acenapthene = 35659
Area of IS = 613275
b = -0.0909161
m^1 = 9.605304
m^2 = 7.132688
ηg of IS = 250
response = 35659/613275 = 0.058145

Amount in ηg on column =

$$(250\eta g)\left[-0.0909161 + 9.605304\ x\ 0.058145 + 7.132688\ x\ 0.058145\text{^}2\right] = 122.9\eta g$$

$$Concentration\ (water)\mu g / L = \frac{(y)(Df)(Vt)}{(Vo)(Vi)}$$

$$Concentration\ (soil)\mu g / Kg = \frac{(y)(Df)(Uf)(Vt)}{(Vi)(Ws)(D)}$$

where: Uf = unit of correction for GPC, if used

12.7.4 Concentration of soil samples (dry weight basis) by GC/MS using linear regression analysis:

$$\frac{A_s C_{is}}{A_{is}} = aC_s + b$$

$$C_s = \frac{\left[\frac{A_s C_{is}}{A_{is}} - b\right]}{a}$$

where:
As = Area of the target analyte peak in the sample
Ais = Area of the internal standard peak
Cs = Concentration of the target analyte in on column the calibration standard
Cis = Concentration of the internal standard
a = Slope of the line (coefficient of Cs)
b = The intercept

$$Concentration\ (\text{water})\mu g / L = \frac{(Cs)(Df)(Vt)}{(Vo)(Vi)}$$

$$Concentration\ (\text{soil})\ \mu g / Kg = \frac{(Cs)(Df)(Uf)(Vt)}{(Vi)(Ws)(D)}$$

12.8 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed by adding 100 µL of sample extract to 900 µL of methylene chloride for a total volume of 1000 µL. It should be recorded on the run log as "10x (1 in 10)."

13.0 <u>Method Performance</u>

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for a single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 8270C, Update IV, 1/08, Method 8270D

16.2 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.3 Quality Control SOP 13.4, "Numerical Data Reduction"

16.4 EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs), EPA/600/B-07/001, April 2007

16.5 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.6 Code of Federal Regulations, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.7 NELAC Standards, effective July 2003, plus revisions

16.8 CompuChem Quality Manual, Revision 13, November 17, 2009, plus revisions

16.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010, plus revisions

17.0 Attachments as Tables, Diagrams, Flowcharts and Validation Data

17.1 Attachment 1 – List of **Analytes**, CAS numbers and key ions

17.2 Attachment 2 – Method Reporting Limits

17.3 Attachment 3 – Example Instrument Run Log

17.4 Attachment 4 – SIM Standards Concentration

Attachment 1

Analyte List with CAS Numbers and Key Ions

Compound	CAS#	Primary Ion	Secondary Ions
Naphthalene	91-20-3	128	129, 127
1-Methylnaphthalene	90-12-0	142	141, 115
2-Methylnaphthalene	91-57-6	142	141, 115
Acenaphthylene	208-96-8	152	151, 153
Acenaphthene	83-32-9	153	154, 152
Fluorene	86-73-7	166	165, 167
Hexachlorobenzene	118-74-1	284	142, 249
Pentachlorophenol	87-86-5	266	264, 268
Phenanthrene	208-96-8	152	151, 153
Anthracene	83-32-9	153	154, 152
Fluoranthene	86-73-7	166	165, 167
Pyrene	129-00-0	202	200, 203
Benzo(a)anthracene	56-55-3	228	226, 229
Chrysene	218-01-9	228	226, 229
Benzo(b)fluoranthene	205-99-2	252	126, 125
Benzo(k)fluoranthene	207-08-9	252	126, 125
Benzo(a)pyrene	50-32-8	252	126, 125
Indeno(1,2,3-cd)pyrene	193-39-5	276	138, 227
Dibenzo(a,h)anthracene	53-70-3	278	139, 279
Benzo(g,h,i)perylene	191-24-2	276	138, 277
4-Nitrophenol	100-02-7	109	65, 139
Hexachlorobutadiene	87-68-3	225	227, 223
1,4-Dichlorobenzene-d4*	2199-69-1	152	115
Naphthalene-d_8 *	1146-65-2	136	68
Acenaphthene-d_{10} *	15067-26-2	164	162, 160
Phenanthrene-d_{10} *	1517-22-2	188	94, 80
Chrysene-d_{12} *	1719-03-5	240	120, 236
Perylene-d_{12} *	1520-96-3	264	260, 265
Nitrobenzene-d_5 $	4165-60-0	82	128, 54
2-Fluorobiphenyl $	321-60-8	172	171, 174
Terphenyl-d_{14} $	98904-43-9	244	122, 212
2,4,6-Tribromophenol $	118-79-6	330	141, 332

* = Internal Standards $ = Surrogate

NOTE: For SIM analyses only one secondary ion is used for identification.

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Attachment 2

Compound	Aqueous Reporting Limit µg/L	Soil Reporting Limit µg/Kg
1-Methylnaphthalene	0.20	8.3
2-Methylnaphthalene	0.20	8.3
4-Nitrophenol	1.9	64
Acenaphthene	0.20	8.3
Acenaphthylene	0.20	8.3
Anthracene	0.20	8.3
Benzo(a)anthracene	0.20	8.3
Benzo(a)pyrene	0.20	8.3
Benzo(b)fluoranthene	0.20	8.3
Benzo(g,h,i)perylene	0.20	8.3
Benzo(k)fluoranthene	0.20	8.3
Chrysene	0.20	8.3
Dibenzo(a,h)anthracene	0.20	8.3
Fluoranthene	0.20	8.3
Fluorene	0.20	8.3
Hexachlorobenzene	0.64	21.3
Hexachlorobutadiene	0.20	8.3
Indeno(1,2,3-cd)pyrene	0.20	8.3
Naphthalene	0.20	8.3
Pentachlorophenol	0.64	21.3
Phenanthrene	0.20	8.3
Pyrene	0.20	8.3

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Attachment 3

COMPUCHEM a Division of Liberty Analytical Corp
GC/MS SEMIVOLATILE RUN LOG
LOGBOOK 4U(4) 23 (5972hp70)

DATE ___/___/___ INITIAL TIME OF TUNE _____ SHIFT/S (A) ____(B)_____(C)____
TIME TUNE EXPIRES_____
METHOD: 8270C 8270D SOM01.2 (Circle One) or Other: _____SIM: (Y/N)

PREVENTIVE MAINTENANCE_____

	SEQUENCE#	REJECTED	FILENAME	REPORTED	DATE	TIME	CLIENT ID	SDG#	AMOUNT INJECTED	CHEMIST	COMMENTS
1					/ /						
2					/ /						
3					/ /						
4					/ /						
5					/ /						
6					/ /						
7					/ /						
8					/ /						
9					/ /						
10					/ /						
11					/ /						
12					/ /						
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19					/ /						
20					/ /						
21					/ /						
22					/ /						
23					/ /						
24					/ /						

DFTPP Lot #/Exp. Date: _____ Calibration STD Lot#/Exp. Date: _____ Internal STD Lot#/Exp. Date: _____ Methylene Chloride Lot#/Exp. Date: _____ Column Type: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

REVIEWED BY: _____ DATE _____

7/9/10:jad

3301

Note: This page subject to change without notice.

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Attachment 4

SIM standards concentration (µg/mL)

Compound/ Compound Group	Level 1	Level 2	Level 3	Level **4**	Level **5**
PAHs	0.2	0.6	1.5	2.5	5.0
Hexachlorobutadiene	0.2	0.6	1.5	2.5	5.0
B/N Surrogates	0.2	0.6	1.5	2.5	5.0
Acid Surrogate	0.4	1.2	3.0	5.0	10.0
4-Nitrophenol	1.0	2.0	4.0	8.0	10.0
Pentachlorophenol	1.0	2.0	4.0	8.0	10.0
Hexachlorobenzene	1.0	2.0	4.0	8.0	10.0

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SOP 2.5.2.1

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP SOP Section #: 2.5.2.1 Revision #: 15

SOP Title: Preparation of Water Samples for the Analysis of Semivolatiles by SW-846 Method 3510C

Effective date: (QA fills in) 10/26/11

- Procedure prepared by: Judith Dozier Date: 10/26/2011
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 10/26/11
- Reason for change: Annual review; change Spike & Surrogate names and lists
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Ed. Update III 12/96, Method 3510C & Method 8270C, Update IV Method 8270D, 2/07; DoD QSM, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure

Preparation of Water Samples for the Analysis of Semivolatiles by SW-846 Method 3510C

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1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the extraction of semivolatile organic compounds from water samples prior to GC/MS analysis.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies. See the Quality Manual for demonstration of capability procedures.

2.0 Summary of Method

A 1 liter volume of sample is spiked with the semivolatile surrogate solution, and acid and base/neutral fractions are extracted by separatory funnel with methylene chloride. The extracts are dried, combined, concentrated, and submitted for GC/MS analysis. Alternatively, acid/neutral and base fractions may be generated separately. This procedure is applicable to the preparation of water samples for analysis by full-scan GC/MS and GC/MS in the Selected Ion Monitoring (SIM) mode.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL

must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – µg/L

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch,

method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 DoD-QSM – Department of Defense Quality Systems Manual

3.10 Extraction Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.11 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Method interferences may be caused by contaminants in reagents, solvents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free of interferences under the conditions of the analysis by preparing and analyzing laboratory reagent blanks.

4.2 Matrix interferences may be caused by contaminants that were inadvertently co-extracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Matrix spike/matrix spike duplicate (MS/MSD) analyses will be done to determine the possible matrix interferences.

4.2.1 For sample extracts demonstrating matrix interferences, gel permeation cleanup procedure, Method 3640A, is an option.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 2 liter separatory funnels (glass or Teflon®) with stopper (or screw cap) and stopcock

6.2 1 liter graduated cylinders or graduated glass jars

6.3 Erlenmeyer flasks

6.4 Powder funnels

6.5 Kuderna-Danish apparatus

6.5.1 Concentrator tubes, 10 mL graduated, Kontes K570050-1025 or equivalent

6.5.2 Evaporative flasks, 500 mL, Kontes K-57001-0500 or equivalent

6.5.3 Snyder column, three ball macro, Kontes K-503000-0121 or equivalent

6.5.4 Snyder column, two ball micro, Chemglass CC-0309-031-MS or equivalent

6.6 Glass stirring rods

6.7 Boiling chips - silicon carbide, furnaced at 400 °C for 30 minutes; or soxhlet extract with methylene **chloride** for 4 hours

6.8 Rubber bands

6.9 Nitrogen evaporation device (Organomation, or equivalent)

6.10 pH paper - wide range

6.11 Glass wool

6.11.1 Furnace at 400 °C for 4 hours prior to use

6.12 Syringes - 100 µL, 250 µL, 500 µL, 1000 µL (1 mL)

6.13 Amber 2-mL vial with a Teflon®-lined screw cap

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6.14 Element DataSystem® LIMS computer software by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the standards preparation log book (22F) **or in Promium LIMS**.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2 - 6 ^{o}C in the laboratory when not in use. Stock solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.

Note: All standards and reagents are subject to change in vendor and in concentration. All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria. It is referred to throughout the remainder of this SOP as reagent water.

7.2 Sodium hydroxide solution, 10 N

7.3 Sulfuric acid - concentrated

7.4 Solvents – pesticide residue analysis grade, or equivalent

7.4.1 Acetone

7.4.2 Methanol

7.4.3 Methylene chloride

7.5 Sodium sulfate (ACS) - powdered, anhydrous.

7.5.1 Purify by heating at 400 °C for four hours in a shallow tray. Cool in a desiccator, and store in a glass bottle.

7.6 Surrogate and Spikes

7.6.1 Full Scan Spiking Solutions

7.6.1.1 **8270 Full** Surrogate **Spike** - See Attachment 1.

7.6.1.2 8270 **LCS/MS Spike 1 and 8270 LCS/MS Spike 2** – See Attachment 1.

7.6.1.3 Appendix IX **LCS/MS** Spike 1 and Appendix IX **LCS/MS** Spike 2 - See Attachment 1.

Note: The full scan spiking solutions may contain compounds not normally requested by clients.

7.6.2 SIM Spiking Solutions

7.6.2.1 **8270 SIM** Surrogate **Spike** - See Attachment 2.

7.6.2.2 Bis(2-chloroethyl)ether (BCEE) – See Attachment 2.

7.6.2.3 **NSI** SIM **Mix** (**Q-6605-O**) - See Attachment 2.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Aqueous samples must be extracted within 7 days of sampling.

8.3 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank is prepared with each extraction batch of up to 20 samples.

9.2 Laboratory Control Sample

9.2.1 A Laboratory Control Sample (LCS) must be prepared with each extraction batch of up to 20 samples.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared with each batch of up to 20 samples.

9.4 Contingency

9.4.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.4.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.4.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration and Standardization

N/A

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 Preparation of Equipment

11.1.1 Cover all work areas with plastic-backed, absorbent table covering, with the plastic side down.

11.1.2 Assemble the following glassware for each sample to be processed:

11.1.2.1 2-liter glass or Teflon® separatory funnel with stopper (or screw-cap) and stopcock

11.1.2.2 1-liter graduated cylinder or graduated glass jar

11.1.2.3 Erlenmeyer flask

11.1.2.4 Powder funnel

11.1.2.5 Kuderna-Danish apparatus

11.1.2.6 Glass stirring rod

11.1.3 Rinse each of the items listed above with methylene chloride. Empty the methylene chloride into a solvent waste container.

11.1.4 Add a small plug of furnaced glass wool to each powder funnel; add 1-2 inches of prepared sodium sulfate to each funnel.

11.1.5 Label each piece of glassware with the sample number.

11.2 Sample Preparation and Extraction

11.2.1 Allow the sample to equilibrate to room temperature. Shake the sample container to mix the sample.

11.2.2 Using a 1 liter graduated cylinder or graduated glass jar, measure 1 liter (nominal) of sample. Record the initial volume on the worksheet. (Attachment 3)

11.2.3 Pour the sample into its appropriately labeled separatory funnel.

11.2.4 Use 5 to 10 mL of methylene chloride to rinse the graduated cylinder or graduated glass jar used to measure the sample. Add the rinse to the separatory funnel.

11.2.4.1 If the entire contents of the sample container are used, use 5 to 10 mL of methylene chloride to rinse the sample container. Add the rinse to the separatory funnel.

11.2.5 Add 0.5 mL of **8270 Full Surrogate Spike** (or **8270 SIM Surrogate Spike**) to each field sample using the syringe. It is important to add exactly 0.5 mL, since surrogate recoveries are used to judge the efficiency of the extraction.

11.2.6 Prepare a method blank by adding 0.5 mL of **8270 Full Surrogate Spike** (or **8270 SIM Surrogate Spike**) to 1000 mL of reagent water.

11.2.7 Prepare the LCS by adding 0.5 mL of appropriate full scan or SIM spiking solution, and **8270 Full Surrogate Spike** (or **8270 SIM Surrogate Spike**) to 1000 mL of reagent water. Appropriate full scan or SIM spiking solutions are designated on the extraction worksheets. (See Attachments 1 or 2.)

11.2.8 Prepare the MS/MSD by adding 0.5 mL each of appropriate full scan or SIM spiking solution, and **8270 Full Surrogate Spike** (or **8270 SIM Surrogate Spike**) to 1000 mL aliquots of the designated field sample. Appropriate full scan or SIM spiking solutions are designated on the extraction worksheets. (See Attachments 1 or 2.)

11.2.9 Record the standard ID numbers, the lot numbers, and volume added on the extraction worksheet for all of the standards used in this procedure.

11.2.10 Slowly add approximately 0.5 mL of concentrated sulfuric acid to each separatory funnel using a serological pipet. Stopper the separatory funnel and shake for 20-30 seconds to mix, venting the stopcock several times.

11.2.11 Check the pH by inserting a disposable pipette into the sample and touching the pipette to a piece of wide range pH paper. If the pH is 2 or less, note the pH on the extraction worksheet. If the pH is > 2, continue to add acid 1 mL at a time, mixing between additions, until the pH has reached 2 or less. The sample is now ready for the acid-neutral extraction.

11.2.12 Add 60 mL of methylene chloride to the separatory funnel. Stopper each funnel and shake vigorously for 2 minutes. Be careful to vent the stopcock frequently under the hood, until the pressure equalizes.

11.2.13 Allow each separatory funnel to hang undisturbed for approximately 10 minutes, to allow the layers to separate. If an emulsion larger than two-thirds the size of the bottom layer (methylene chloride) forms, steps must be taken to break it up. Emulsions may be broken by stirring, passing through the stopcock very slowly, or by centrifugation. The method used is determined by the severity of the emulsion.

11.2.13.1 To break an emulsion using centrifugation, drain the emulsion into a centrifuge bottle. Place the bottle in the centrifuge. (Always use a bottle with approximately the sample volume of methylene chloride as a counter-weight, if

only one sample is being centrifuged). Secure the lid of the centrifuge and start. Let the sample(s) spin for 2 minutes at 2000 RPM and turn off the centrifuge. Once the sample(s) have stopped spinning, remove the bottles. Pour the sample extract back into the appropriate separatory funnel. Rinse the bottle with 5 to 10-mL of methylene chloride and pour the rinse into the separatory funnel.

11.2.14 When two distinct layers are obtained, drain the lower layer (methylene chloride) through a powder funnel containing glass wool and sodium sulfate into the Erlenmeyer flask. Close the stopcock when the water layer reaches the stopcock. The object is to collect all of the methylene chloride and none of the water.

11.2.15 Add 60 mL of methylene chloride to the separatory funnel. Repeat the steps in **Sections** 11.2.12 through 11.2.14 two more times.

11.2.16 Slowly add approximately **5.0** mL of 10 N sodium hydroxide to each separatory funnel. Stopper the separatory funnel and shake for 20-30 seconds, venting the stopcock under the hood several times.

11.12.17 Check the pH by inserting a disposable pipette into the sample and touching the pipette to a piece of wide range pH paper. If the pH is now 11 or greater, note the pH on the extraction worksheet. If the pH is not 11 or greater, continue to add 1 mL volumes of sodium hydroxide. Shake and measure the pH until a value of 11 or greater is reached.

11.2.18 Add 60 mL of methylene chloride to the separatory funnel. Repeat the steps in Sections 11.2.12 through 11.2.14 two more times.

11.2.19 Pour the combined acid-neutral and base extracts from the Erlenmeyer flask into the K-D flask and transfer the sample label.

11.3 Extract Concentration

11.3.1 Add 2-3 boiling chips to each K-D flask and attach a Snyder column. Add 1-2-mL of methylene chloride to the top of the Snyder column.

11.3.2 Place the K-D apparatus on a water bath set at 85-95 °C. Adjust the vertical position of the apparatus and water temperature as required to concentrate the extract in 15-30 minutes.

11.3.3 Remove each K-D flask from the bath as soon as an apparent volume of 4 mL is reached and allow it to drain.

11.4 Final Extract Concentration

11.4.1 Remove the Snyder column and evaporation flask from the concentrator tube.

11.4.2 Place a 2-ball micro Snyder column on the concentration tube. Wet the micro column by placing approximately 1 mL of methylene chloride into the top of the column.

11.4.1.1 Hold the concentrator tube over the hot bath and concentrate the extract to an apparent volume of 0.5 mL.

11.4.1.2 Remove concentrator tube from the bath and allow it to cool and drain.

11.4.3 Alternately, place the concentrator tube containing the extract into the nitrogen concentration device.

11.4.1.3 Using a clean, dry stream of nitrogen, concentrate the extract to approximately 0.5 mL.

11.4.4 Quantitatively transfer the entire extract volume with a transfer pipette to an amber, 2 mL autosampler vial. Rinse the concentrator tube with approximately 0.5 mL of methylene chloride and add the rinse to the sample vial. Adjust the final extract volume to 1.0 mL and mark the extract volume on the vial with a permanent marker.

11.4.5 Label the vial with CompuChem number, and extraction date. At the time of transfer, note the final volume on the Extraction worksheet.

11.4.6 Complete the paperwork. Record the lot numbers for all reagents used in this procedure on the extraction worksheet. Give the completed worksheet to the supervisor or his/her designee for review and signature.

11.4.7 Deliver the sample extracts and copies of the completed worksheets to the semivolatile GC/MS lab for analysis, under Chain-of-Custody.

12.0 <u>Data Analysis and Calculations</u>

N/A

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96, Methods 3510C and 8270C, and Update IV Method 8270D, 02/07

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.4 **TNI** Standards, approved **July 2011**, plus revisions

16.5 QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.6 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.7 Sample Control SOP 4.6, "Storing Samples"

16.8 Department of Defense Quality Systems Manual of Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – Sample Full Scan Spiking Solution Worksheets

17.2 Attachment 2 - Sample SIM Spiking Solution Worksheets

17.3 Attachment 3 – **Example** Extraction Worksheet

Attachment 1

Analytical Standard Record
COMPUCHEM
1J18003

Description:	8270 LCS/MS Spike 1	Expires:	04/18/2012
Standard Type:	Analyte Spike	Prepared:	10/18/2011
Solvent:	MeOH/DE464	Prepared By:	Brad Lake
Final Volume (mls):	50	Department:	SVOA
Vials:	1	Last Edit:	10/21/2011 11:45 by BWL

65378 and co-elution factor

Analyte	CAS Number	Concentration	Units
4-Chloro-3-methylphenol	59-50-7	80	ug/mL
2-Methylnaphthalene	91-57-6	80	ug/mL
2-Methylphenol	95-48-7	80	ug/mL
2-Nitroaniline	88-74-4	80	ug/mL
2-Nitrophenol	88-75-5	80	ug/mL
3 & 4-Methylphenol	106-44-5	80	ug/mL
3-Methylphenol	108-39-4	80	ug/mL
3-Nitroaniline	99-09-2	80	ug/mL
Aniline	62-53-3	80	ug/mL
4-Bromophenyl-phenylether	101-55-3	80	ug/mL
2,6-Dinitrotoluene	606-20-2	80	ug/mL
4-Chloroaniline	106-47-8	80	ug/mL
4-Chlorophenyl-phenylether	7005-72-3	80	ug/mL
4-Methylphenol	106-44-5	80	ug/mL
4-Nitroaniline	100-01-6	80	ug/mL
4-Nitrophenol	100-02-7	80	ug/mL
Acenaphthene	83-32-9	80	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	80	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	80	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	80	ug/mL
1,2,4-Trichlorobenzene	120-82-1	80	ug/mL
1,2-Dichlorobenzene	95-50-1	80	ug/mL
1,2-Dinitrobenzene	528-29-0	80	ug/mL
1,3-Dichlorobenzene	541-73-1	80	ug/mL
1,3-Dinitrobenzene	99-65-0	80	ug/mL
1,4-Dichlorobenzene	106-46-7	80	ug/mL
1,4-Dinitrobenzene	100-25-4	80	ug/mL
2-Chlorophenol	95-57-8	80	ug/mL
2,2'-oxybis(1-Chloropropane)	108-60-1	80	ug/mL
2-Chloronaphthalene	91-58-7	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1J18003

2,3,5,6-Tetrachlorophenol	935-95-5	80	ug/mL
2,4,5-Trichlorophenol	95-95-4	80	ug/mL
2,4,6-Trichlorophenol	88-06-2	80	ug/mL
2,4-Dichlorophenol	120-83-2	80	ug/mL
2,4-Dimethylphenol	105-67-9	80	ug/mL
2,4-Dinitrophenol	51-28-5	80	ug/mL
2,4-Dinitrotoluene	121-14-2	80	ug/mL
Anthracene	120-12-7	80	ug/mL
1-Methylnaphthalene	90-12-0	80	ug/mL
N-Nitrosodimethylamine	62-75-9	80	ug/mL
Fluoranthene	206-44-0	80	ug/mL
Fluorene	86-73-7	80	ug/mL
Hexachlorobenzene	118-74-1	80	ug/mL
Hexachlorobutadiene	87-68-3	80	ug/mL
Hexachlorocyclopentadiene	77-47-4	80	ug/mL
Hexachloroethane	67-72-1	80	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	80	ug/mL
Acenaphthylene	208-96-8	80	ug/mL
N-Nitroso-di-N-propylamine	621-64-7	80	ug/mL
Dibenzofuran	132-64-9	80	ug/mL
N-Nitrosodiphenylamine (1)	86-30-6	80	ug/mL
Naphthalene	91-20-3	80	ug/mL
Nitrobenzene	98-95-3	80	ug/mL
Pentachlorophenol	87-86-5	80	ug/mL
Phenanthrene	85-01-8	80	ug/mL
Phenol	108-95-2	80	ug/mL
Pyrene	129-00-0	80	ug/mL
Isophorone	78-59-1	80	ug/mL
bis(2-ethylhexyl)Phthalate	117-81-7	80	ug/mL
Azobenzene	103-33-3	80	ug/mL
Benzo (a) anthracene	56-55-3	80	ug/mL
Benzo (a) pyrene	50-32-8	80	ug/mL
Benzo (b) fluoranthene	205-99-2	80	ug/mL
Benzo (g,h,i) perylene	191-24-2	80	ug/mL
Benzo(k)fluoranthene	207-08-9	80	ug/mL
Benzyl alcohol	100-51-6	80	ug/mL
Dimethylphthalate	131-11-3	80	ug/mL
Bis(2-chloroethyl)ether	111-44-4	80	ug/mL
Diethylphthalate	84-66-2	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1J18003

Bis-(2-Ethylhexyl) Adipate	103-23-1	80	ug/mL
Butylbenzylphthalate	85-68-7	80	ug/mL
Carbazole	86-74-8	80	ug/mL
Chrysene	218-01-9	80	ug/mL
Di-n-butylphthalate	84-74-2	80	ug/mL
Di-n-octylphthalate	117-84-0	80	ug/mL
Dibenzo(a,h)anthracene	53-70-3	80	ug/mL
Pyridine	110-86-1	80	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	80	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0F30010	3 & 4-Methylphenol CO-ELUTION	06/30/2010	Jaime Redig	04/04/2015	12/09/2010 10:33 by jer	2
0K24005	3 & 4-Methylphenol	11/24/2010	Ken Grzybowski	11/19/2015	01/14/2011 08:56 by jer	4
1G15005	NSI 8270 BNA Mix (C-701)	07/15/2011	** Vendor **	05/30/2013	07/18/2011 12:01 by BWL	4
1H03001	Accustandard 1,2,4,5-Tetrachlorob	08/03/2011	** Vendor **	06/10/2021	08/30/2011 16:45 by BWL	4

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1J18004

Description:	8270 LCS/MS Spike 2	Expires:	04/18/2012
Standard Type:	Analyte Spike	Prepared:	10/18/2011
Solvent:	MeOH/DE464	Prepared By:	Brad Lake
Final Volume (mls):	50	Department:	SVOA
Vials:	1	Last Edit:	10/18/2011 11:50 by BWL

65379

Analyte	CAS Number	Concentration	Units
Caprolactam	105-60-2	80	ug/mL
Benzidine	92-87-5	80	ug/mL
Benzaldehyde	100-52-7	80	ug/mL
Atrazine	1912-24-9	80	ug/mL
Acetophenone	98-86-2	80	ug/mL
3,3'-Dimethylbenzidine	119-93-7	80	ug/mL
3,3'-Dichlorobenzidine	91-94-1	80	ug/mL
1,1'-Biphenyl	92-52-4	80	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
1H02009	Absolute SV OLMO4.2 Additional	08/02/2011	** Vendor **	04/12/2016	08/30/2011 17:04 by BWL	2
1H02024	Restek 8270 Benzidines Mix #2 (3	08/02/2011	Brad Lake	04/30/2019	08/30/2011 17:12 by BWL	2

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1G13014

Description:	APP IX Spike I	Expires:	12/31/2011
Standard Type:	Analyte Spike	Prepared:	07/13/2011
Solvent:	MeOH/DD151	Prepared By:	Brad Lake
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	09/22/2011 15:56 by SLP

65145

Analyte	CAS Number	Concentration	Units
4-Nitroquinoline-1-oxide	56-57-5	80	ug/mL
Dibenz (a,j) acridine	224-42-0	80	ug/mL
Diallate (trans)	17708-58-6	80	ug/mL
Diallate (cis)	17708-57-5	80	ug/mL
Diallate	2303-16-4	80	ug/mL
Chlorobenzilate	510-15-6	80	ug/mL
Caprolactam	105-60-2	80	ug/mL
Biphenyl	92-52-4	80	ug/mL
Benzidine	92-87-5	80	ug/mL
Benzaldehyde	100-52-7	80	ug/mL
Atrazine	1912-24-9	80	ug/mL
Aramite	140-57-8	80	ug/mL
Acetophenone	98-86-2	80	ug/mL
a,a-Dimethylphenethylamine	122-09-8	80	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	80	ug/mL
2-Naphthylamine	91-59-8	80	ug/mL
1,3,5-Trinitrobenzene	99-35-4	80	ug/mL
1,4-Dioxane	123-91-1	80	ug/mL
1,4-Naphthoquinone	130-15-4	80	ug/mL
1-Chloronaphthalene	90-13-1	80	ug/mL
1-Naphthylamine	134-32-7	80	ug/mL
7,12-Dimethylbenz(a)anthracene	57-97-6	80	ug/mL
2-Acetylaminofluorene	53-96-3	80	ug/mL
5-Nitro-o-toluidine	99-55-8	80	ug/mL
2-Picoline	109-06-8	80	ug/mL
3,3'-Dichlorobenzidine	91-94-1	80	ug/mL
3,3'-Dimethylbenzidine	119-93-7	80	ug/mL
3-Methylcholanthrene	56-49-5	80	ug/mL
4-Aminobiphenyl	92-67-1	80	ug/mL
Disulfoton	298-04-4	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1G13014

2,6-Dichlorophenol	87-65-0	80	ug/mL
Pentachloroethane	76-01-7	80	ug/mL
Dimethoate	60-51-5	80	ug/mL
o,o,o-Triethyl phosphorothioate	126-68-1	80	ug/mL
o-Toluidine	95-53-4	80	ug/mL
p-Dimethylaminoazobenzene	60-11-7	80	ug/mL
p-Phenylenediamine	106-50-3	80	ug/mL
N-Nitrosopiperidine	100-75-4	80	ug/mL
Pentachlorobenzene	608-93-5	80	ug/mL
N-Nitrosomorpholine	59-89-2	80	ug/mL
Pentachloronitrobenzene	82-68-8	80	ug/mL
Phenacetin	62-44-2	80	ug/mL
Phorate	298-02-2	80	ug/mL
Pronamide	23950-58-5	80	ug/mL
Safrole	94-59-7	80	ug/mL
Sulfotepp	3689-24-5	80	ug/mL
Parathion	56-38-2	80	ug/mL
Kepone	143-50-0	80	ug/mL
Zinophos	297-97-2	80	ug/mL
Ethyl Methacrylate	97-63-2	80	ug/mL
Ethyl Methanesulfonate	62-50-0	80	ug/mL
Famphur	52-85-7	80	ug/mL
Hexachloropropene	1888-71-7	80	ug/mL
N-Nitrosopyrrolidine	930-55-2	80	ug/mL
Isosafrole	120-58-1	80	ug/mL
Diphenyl ether	101-84-8	80	ug/mL
Methapyrilene	91-80-5	80	ug/mL
Methyl Methanesulfonate	66-27-3	80	ug/mL
Methyl Parathion	298-00-0	80	ug/mL
N-Nitroso-di-n-butylamine	924-16-3	80	ug/mL
N-Nitrosodiethylamine	55-18-5	80	ug/mL
N-Nitrosomethylethylamine	10595-95-6	80	ug/mL
Isodrin	465-73-6	80	ug/mL

Reviewed By _____ Date _____

Page 2 of 3

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1G13014

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0L23023	Restek@32419 8270 OP Pest Mix	12/23/2010	** Vendor **	01/31/2012	12/23/2010 12:55 by jer	1
1G13009	Restek APP IX Mix #1 (31625)	07/13/2011	** Vendor **	12/31/2011	09/22/2011 15:56 by SLP	1
1G13011	Restek Benzidine Mix (31834)	07/13/2011	** Vendor **	03/31/2019	07/18/2011 11:32 by BWL	1
1G13012	Restek APP IX Mix #2 (31806)	07/13/2011	** Vendor **	09/30/2012	08/17/2011 15:07 by KG	2

Reviewed By Date

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1G13013

Description:	APP IX Spike II	Expires:	01/14/2012
Standard Type:	Analyte Spike	Prepared:	07/13/2011
Solvent:	MeOH/DD151	Prepared By:	Brad Lake
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	09/22/2011 15:57 by SLP

65146

Analyte	CAS Number	Concentration	Units
4-Chloro-3-methylphenol	59-50-7	80	ug/mL
2-Methylnaphthalene	91-57-6	80	ug/mL
2-Methylphenol	95-48-7	80	ug/mL
2-Nitroaniline	88-74-4	80	ug/mL
2-Nitrophenol	88-75-5	80	ug/mL
3 & 4-Methylphenol	106-44-5	80	ug/mL
3-Methylphenol	108-39-4	80	ug/mL
3-Nitroaniline	99-09-2	80	ug/mL
Aniline	62-53-3	80	ug/mL
4-Bromophenyl-phenylether	101-55-3	80	ug/mL
2,6-Dinitrotoluene	606-20-2	80	ug/mL
4-Chloroaniline	106-47-8	80	ug/mL
4-Chlorophenyl-phenylether	7005-72-3	80	ug/mL
4-Methylphenol	106-44-5	80	ug/mL
4-Nitroaniline	100-01-6	80	ug/mL
4-Nitrophenol	100-02-7	80	ug/mL
Acenaphthene	83-32-9	80	ug/mL
1,2,4-Trichlorobenzene	120-82-1	80	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	80	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	80	ug/mL
1,2-Dichlorobenzene	95-50-1	80	ug/mL
1,2-Dinitrobenzene	528-29-0	80	ug/mL
1,2-Diphenyl Hydrazine	122-66-7	80	ug/mL
1,3-Dichlorobenzene	541-73-1	80	ug/mL
1,3-Dinitrobenzene	99-65-0	80	ug/mL
1,4-Dichlorobenzene	106-46-7	80	ug/mL
1,4-Dinitrobenzene	100-25-4	80	ug/mL
2-Chlorophenol	95-57-8	80	ug/mL
2,2'-oxybis(1-Chloropropane)	108-60-1	80	ug/mL
2-Chloronaphthalene	91-58-7	80	ug/mL

Reviewed By _____ Date _____

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1G13013

2,3,5,6-Tetrachlorophenol	935-95-5	80	ug/mL
2,4,5-Trichlorophenol	95-95-4	80	ug/mL
2,4,6-Trichlorophenol	88-06-2	80	ug/mL
2,4-Dichlorophenol	120-83-2	80	ug/mL
2,4-Dimethylphenol	105-67-9	80	ug/mL
2,4-Dinitrophenol	51-28-5	80	ug/mL
2,4-Dinitrotoluene	121-14-2	80	ug/mL
Anthracene	120-12-7	80	ug/mL
1-Methylnaphthalene	90-12-0	80	ug/mL
N-Nitrosodimethylamine	62-75-9	80	ug/mL
Fluoranthene	206-44-0	80	ug/mL
Fluorene	86-73-7	80	ug/mL
Hexachlorobenzene	118-74-1	80	ug/mL
Hexachlorobutadiene	87-68-3	80	ug/mL
Hexachlorocyclopentadiene	77-47-4	80	ug/mL
Hexachloroethane	67-72-1	80	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	80	ug/mL
Acenaphthylene	208-96-8	80	ug/mL
N-Nitroso-di-N-propylamine	621-64-7	80	ug/mL
Dibenzofuran	132-64-9	80	ug/mL
N-Nitrosodiphenylamine (1)	86-30-6	80	ug/mL
Naphthalene	91-20-3	80	ug/mL
Nitrobenzene	98-95-3	80	ug/mL
Pentachlorophenol	87-86-5	80	ug/mL
Phenanthrene	85-01-8	80	ug/mL
Phenol	108-95-2	80	ug/mL
Pyrene	129-00-0	80	ug/mL
Isophorone	78-59-1	80	ug/mL
bis(2-ethylhexyl)Phthalate	117-81-7	80	ug/mL
Benzo (a) anthracene	56-55-3	80	ug/mL
Benzo (a) pyrene	50-32-8	80	ug/mL
Benzo (b) fluoranthene	205-99-2	80	ug/mL
Benzo (g,h,i) perylene	191-24-2	80	ug/mL
Benzo(k)fluoranthene	207-08-9	80	ug/mL
Benzoic acid	65-85-0	80	ug/mL
Benzyl alcohol	100-51-6	80	ug/mL
Dimethylphthalate	131-11-3	80	ug/mL
Bis(2-chloroethyl)ether	111-44-4	80	ug/mL
Diethylphthalate	84-66-2	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1G13013

Bis-(2-ethylhexyl)adipate	103-23-1	80	ug/mL
Butylbenzylphthalate	85-68-7	80	ug/mL
Carbazole	86-74-8	80	ug/mL
Chrysene	218-01-9	80	ug/mL
Di-n-butylphthalate	84-74-2	80	ug/mL
Di-n-octylphthalate	117-84-0	80	ug/mL
Dibenzo(a,h)anthracene	53-70-3	80	ug/mL
Pyridine	110-86-1	80	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	80	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0F30010	3 & 4-Methylphenol CO-ELUTION	06/30/2010	Jaime Redig	04/04/2015	12/09/2010 10:33 by jer	1
0J01012	Restek Benzoic Acid 31879	10/01/2010	** Vendor **	06/30/2014	10/01/2010 17:02 by jer	1
0K24005	3 & 4-Methylphenol	11/24/2010	Ken Grzybowski	11/19/2015	01/14/2011 08:56 by jer	2
1G13010	Restek 8270 MegaMix (31850)	07/13/2011	** Vendor **	10/31/2012	09/22/2011 15:57 by SLP	2

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
1F08011

Description:	#393 NSI BNA Surrogates (SW 846) (WL	Expires:	05/31/2013
Standard Type:	Surrogate Spike	Prepared:	06/08/2011
Solvent:	Methanol	Prepared By:	** Vendor **
Final Volume (mls):	25	Department:	SVOA
Vials:	16	Last Edit:	06/13/2011 14:34 by BWL

052311-40

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	98904-43-9	100	ug/mL
Phenol-d5	4165-62-2	200	ug/mL
Nitrobenzene-d5	4165-60-0	100	ug/mL
2-Fluorophenol	367-12-4	200	ug/mL
2-Fluorobiphenyl	321-60-8	100	ug/mL
2,4,6-Tribromophenol	118-79-6	200	ug/mL

Reviewed By _____ Date _____

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Attachment 2

Analytical Standard Record
COMPUCHEM
1F08013

Description:	NSI SIM Mix (Q-6605-O)	Expires:	05/31/2014
Standard Type:	Analyte Spike	Prepared:	06/08/2011
Solvent:	MeOH	Prepared By:	** Vendor **
Final Volume (mls):	25	Department:	SVOA
Vials:	2	Last Edit:	06/13/2011 14:36 by BWL

051211-01

Analyte	CAS Number	Concentration	Units
Chrysene	218-01-9	1	ug/mL
2,4-Dinitrophenol	51-28-5	4	ug/mL
2-Methylnaphthalene	91-57-6	1	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	4	ug/mL
4-Nitrophenol	100-02-7	4	ug/mL
Acenaphthene	83-32-9	1	ug/mL
Acenaphthylene	208-96-8	1	ug/mL
Anthracene	120-12-7	1	ug/mL
Benzo (a) anthracene	56-55-3	1	ug/mL
Benzo (a) pyrene	50-32-8	1	ug/mL
Benzo (b) fluoranthene	205-99-2	1	ug/mL
1-Methylnaphthalene	90-12-0	1	ug/mL
Benzo(k)fluoranthene	207-08-9	1	ug/mL
Pyrene	129-00-0	1	ug/mL
Dibenzo(a,h)anthracene	53-70-3	1	ug/mL
Dibenzofuran	132-64-9	1	ug/mL
Fluoranthene	206-44-0	1	ug/mL
Fluorene	86-73-7	1	ug/mL
Hexachlorobenzene	118-74-1	4	ug/mL
Hexachlorobutadiene	87-68-3	1	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	1	ug/mL
Naphthalene	91-20-3	1	ug/mL
Pentachlorophenol	87-86-5	4	ug/mL
Phenanthrene	85-01-8	1	ug/mL
Benzo (g,h,i) perylene	191-24-2	1	ug/mL

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Attachment 2 (continued)

Analytical Standard Record
COMPUCHEM
1I01008

Description:	BCEE LCS Spike	Expires:	03/02/2012
Standard Type:	Analyte Spike	Prepared:	09/01/2011
Solvent:	Methanol/DD151	Prepared By:	Brad Lake
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	09/23/2011 11:29 by BWL

65242

Analyte	CAS Number	Concentration	Units
Bis(2-chloroethyl)ether	111-44-4	1	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0A14019	Absolute BCEE 70075	01/14/2010	** Vendor **	01/04/2015	09/10/2010 07:21 by jer	0.025

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Attachment 2 (continued)

Analytical Standard Record
COMPUCHEM
1H30002

Description:	8270 SIM Surr. Spike	Expires:	02/28/2012
Standard Type:	Surrogate Spike	Prepared:	08/30/2011
Solvent:	MeOH/DD151	Prepared By:	Brad Lake
Final Volume (mls):	200	Department:	SVOA
Vials:	1	Last Edit:	09/23/2011 11:26 by BWL

65236

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	98904-43-9	1	ug/mL
Phenol-d5	4165-62-2	2	ug/mL
Nitrobenzene-d5	4165-60-0	1	ug/mL
2-Fluorophenol	367-12-4	2	ug/mL
2-Fluorobiphenyl	321-60-8	1	ug/mL
2,4,6-Tribromophenol	118-79-6	2	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
1F08011	#393 NSI BNA Surrogates (SW 8	406/08/2011	** Vendor **	05/31/2013	06/13/2011 14:34 by BWL	2

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Attachment 2 (continued)

Analytical Standard Record
COMPUCHEM
1F08011

Description:	#393 NSI BNA Surrogates (SW 846) (WL	Expires:	05/31/2013
Standard Type:	Surrogate Spike	Prepared:	06/08/2011
Solvent:	Methanol	Prepared By:	** Vendor **
Final Volume (mls):	25	Department:	SVOA
Vials:	16	Last Edit:	06/13/2011 14:34 by BWL

052311-40

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	98904-43-9	100	ug/mL
Phenol-d5	4165-62-2	200	ug/mL
Nitrobenzene-d5	4165-60-0	100	ug/mL
2-Fluorophenol	367-12-4	200	ug/mL
2-Fluorobiphenyl	321-60-8	100	ug/mL
2,4,6-Tribromophenol	118-79-6	200	ug/mL

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Attachment 3

Assigned To _____

Employee ID Number _____

PREPARATION BENCH SHEET

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial	Final	Adjusted pH		QC	Surr (uL)	Comments

	Description	Spike Amount (uL)	Lot Number
SURROGATE			
SPIKE			

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

GPC Batch: _____ GPC RUN Date: _____ GPC CAL Date: _____ GPC INST#: _____ Filter Brand: _____ Filter Lot#: _____

Florisil Batch: _____ Florisil RUN Date: _____ Water Florisil Lot #: _____

Acid Wash Batch: _____ Acid Wash By: _____ Acid Wash Date: _____

Derivatization By: _____ Derivatization Date: _____

Date/Time Started: _____

Date/Time Stopped: _____

Manuf. and lot # of reagents/solvents used: _____

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SOP 2.5.2.3

CompuChem
A Division Of
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501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: -176 SOP Section #: 2.5.2.3 Revision #: 16

SOP Title: Preparation of Soil/Sediment/Sludge Samples by SW-846 Method 3550B and Method 3550C for the Analysis of Low-Level Semivolatiles

Effective date: (QA fills in) 04/17/2012

- Procedure prepared by: Bob Meuen Date: 04/17/2012
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Steven L. Rusch Date: 4/18/12
- Reason for change: Clarify concentrations and preparations of LCS/MS and surrogate spikes and when they are added and annual review
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, method 3550B and method 8270C, 12/96; Update IV, method 3550C and method 8270D, 02/07, DoD QSW, Ver. 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -176: Preparation of Soil/Sediment/Sludge Samples by SW-846 Method 3550B and Method 3550C for the Analysis of Low-Level Semivolatiles

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Sample Preparation Procedure -176: Preparation of Soil/Sediment/Sludge Samples by SW-846 Method 3550B and Method 3550C for the Analysis of Low-Level Semivolatiles

1.0 Scope and Application

This procedure is used to determine the concentration of semivolatile organic compounds in solid samples following the procedure in SW-846 (Methods 3550B and 3550C).

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A 30 gram aliquot of sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using sonication. The extract is separated from the sample by Buchner funnel filtration. The sample is concentrated for analysis by GC/MS. Optional extract cleanup procedures may be employed. This procedure is applicable to the preparation of soil samples for analysis by full-scan GC/MS and GC/MS in the Selected Ion Monitoring (SIM) mode.

This procedure encompasses both EPA Methods 3550B and 3550C. All samples for SC DHEC must be processed under EPA 3550C. This SOP is NOT for use with Ohio VAP projects. Please see Ohio VAP specific SOP.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B). A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL

must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.5.3 The reporting limit for CLP is the Contract Required Quantitation Limit (CRQL) for organics.

3.6 Reporting Units – µg/Kg

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5%. If samples are batched together from different sites, project-specific QC must be processed.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 DoD-QSM – Department of Defense-Quality Systems Manual

3.10 Extraction Batch – a group of to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.11 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Method interferences may be caused by contaminants in reagents, solvents, glassware, and other sample-processing hardware that lead to discrete artifacts or elevated baselines in the total ion current profiles (TICPs). All of these materials must be routinely demonstrated to be free of interferences under the conditions of the analysis by preparing and analyzing laboratory reagent blanks.

4.2 Matrix interferences may be caused by contaminants that were inadvertently coextracted from the sample. The extent of matrix interferences will vary considerably from sample to sample. Matrix spike/matrix spike duplicate (MS/MSD) analyses will be done to determine the possible matrix interferences.

5.0 Safety

5.1 The degree of toxicity or carcinogenicity of the chemicals used in this method has not been determined. Each chemical should be treated as a potential health hazard, and exposure to these chemicals should be minimized. Each analyst is responsible for ensuring staff awareness of Occupational Safety and Health Administration (OSHA) regulations regarding safe handling of chemicals used in this method. Additional material on laboratory safety is available for the analyst.

5.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens:

- benzo(a)anthracene
- benzidine
- 3,3′-dichlorobenzidine
- benzo(a)pyrene
- dibenzo(a,h)anthracene
- N-nitrosodimethylamine

5.3 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.4 Laboratory staff members are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Ultrasonic Disrupter, having a minimum power wattage of 300 watts, with pulsing capability. Use a 3/4 "horn."

6.2 Sonabox, to be used with disruptor for decreasing cavitation sound.

6.3 Disposable, graduated, transfer pipets- 0.5 mL, 1.0 mL.

6.4 Teflon® beakers – 250 mL.

6.5 Powder Funnel - 10 cm. diameter with Munktell grade 389 filter paper, or Buchner funnel, or equivalent.

6.6 Kuderna-Danish (K-D) apparatus.

6.6.1 Concentration tube- 10 mL, graduated.

6.6.2 Evaporation flask- 500 mL, attached to concentrator tube with springs or clamps.

6.6.3 Snyder column- three ball macro.

6.7 Boiling chips- solvent extracted (silicon carbide or equivalent).

6.7.1 Heat the chips to 400 °C for 30 minutes prior to use. If no heated boiling chips are available, rinse new boiling chips with methylene chloride before use.

6.8 Water bath- Heated, capable of temperature control (± 5°C)

6.9 Balance- Platform, capable of weighing to nearest 0.01 g.

6.10 Nitrogen blow-down apparatus-Organomation or equivalent.

6.11 Vials- 2 mL, with screw-top, Teflon-lined septa.

6.12 Spatula- Puritan, Standard Tongue Depressors.

6.13 Syringes – 1.0 mL or 2.0 mL (optional)

6.14 Element DataSystem® LIMS by Promium

7.0 Reagents and Standards

Details for the standard preparation are contained in the Standards Preparation Logbook (22F) or Promium Element® LIMS. **See Attachments 1 and 2 to this SOP for the standard concentrations of the surrogate, LCS, and MS spiking solutions used in this method and the preparation information.**

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard

you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

All standards are prepared in the Organic Standards Laboratory. Standards are stored separately from samples at 2-6 °C in the laboratory when not in use. Stock solutions must be prepared every six months or sooner if comparisons with quality control check samples indicate degradation or concentration of solution compounds.

Note: All standards and reagents are subject to change in vendor and in concentration. Standards may be purchased as certified neat materials or concentrated solutions. All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type I water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 Methylene chloride - pesticide-grade methylene chloride is used for this analysis.

7.3 Acetone - pesticide-grade

7.4 Use methylene chloride and acetone to make a 50/50 solution.

7.5 Sodium sulfate - Use only sodium sulfate labeled "FURNACED SODIUM SULFATE." Furnace the sodium sulfate in a shallow tray prior to use for at least four hour in a 400 °C oven.

7.6 Surrogate and Spikes

7.6.1 Full Scan Spiking Solutions

7.6.1.1 Surrogate Spiking Solution #393 - See Attachment 1.

7.6.1.2 8270 Validation I and 8270 Validation II– See Attachment 1.

7.6.1.3 8270 Appendix IX Spike 1 and Appendix IX Spike 2 - See Attachment 1.

Note: The full scan spiking solutions may contain compounds not normally requested by clients.

7.6.2 SIM Spiking Solutions

7.6.2.1 Surrogate Spiking Solution DIL #393 - See Attachment 2.

7.6.2.2 8270 SIM (NSI SIM Mix Q-6605-O) - See Attachment 2.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOPs 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be extracted within 14 days of collection.

8.3 Prior to analysis, all extracts must be stored under refrigeration at 2-6 °C. To meet the requirements of the DoD-QSM, extracts must be stored in the freezer at -10 °C in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Method Blank

9.1.1 A method blank must be prepared with each extraction batch of up to 20 samples.

9.2 Laboratory Control Sample

9.2.1 A laboratory control sample (LCS, or blank spike, BS,) must be prepared with each extraction batch of up to 20 samples.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 A matrix spike and matrix spike duplicate (MS/MSD) are prepared for every sample delivery group (SDG)/ extraction batch.

9.4 Contingency

9.4.1 If due to a lab accident or to QC failure a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.4.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.4.3 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 Ensure the balance has been calibrated for the day prior to its use following the Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. The sample preparation technician must complete the extraction bench sheet (Attachment 1). Any unused portions must be z'ed out. The laboratory supervisor reviews the completed bench sheet for accuracy and completeness and then signs it. The bench sheet accompanies the sample to the analytical laboratory. Include on the bench sheet the manufacturer and lot number of the reagents/solvents used.

11.1 Preparation of Equipment

11.1.1 Rinse each of the items listed above with methylene chloride. Empty the methylene chloride into a waste container and repeat the process two more times. If the glassware selected for use is wet, it must be rinsed with methanol before the methylene chloride rinses.

11.1.2 Label each piece of glassware with the sample number it is to be used for and the method (in this case -8270). Use orange labels.

11.2 Sample Preparation and Extraction

11.2.1 Decant and discard any water layer on a sediment sample. Mix thoroughly to achieve homogeneity. Discard any foreign objects such as sticks, stones, and leaves. Samples should be at room temperature. Place a 250 mL Teflon® beaker on the platform balance and press the tare button. This will automatically subtract the weight of the beaker. Using a spatula, weigh 30.0 g (± 0.1 g) of the sample in the beaker and record the exact weight of the sample to one decimal place on the extraction preparation bench sheet (Attachment 3).

11.2.2 Two additional 30 g portions from a designated sample are transferred to separate beakers for the matrix spike and matrix spike duplicate.

11.2.3 For the blank and LCS, weigh 90.0 g of sodium sulfate.

11.2.4 Add 60 g of furnaced sodium sulfate to each sample. Mix thoroughly to form a free-flowing powder. Additional sodium sulfate may be added if necessary to form a free-flowing powder.

11.2.5 Add 0.5 mL of surrogate standard #393 (or dilute #393 for SIM analyses) to each field sample with a 0.5 mL or 1.0 mL graduated pipet. Record the surrogate standard ID number, the lot number, and volume added on the extraction preparation bench sheet.

Note: For Method 3550C, the surrogate spike and validation spikes (for the LCS and MS/MSD) are **supposed to be** added prior to the addition of the sodium sulfate. **In a memo from the Office of Resource Conservation and Recovery (ORCR), dated August 5, 2010, with the Subject "Spiking (Prior To vs. After Sample Drying) Issue in SW-846 Organic Extraction Methods," EPA recommended not to add spiking solutions prior to the addition of the sodium sulfate. We will follow those recommendations.**

11.2.6 Add 0.5 mL of appropriate full scan or SIM spiking solution and surrogate standard #393 (or dilute #393 for SIM analyses) to the MS/MSD and LCS. Record the standard ID numbers, the lot numbers, and volume added on the extraction preparation bench sheet. Appropriate full scan or SIM spiking solutions are designated on the extraction worksheets. (See Attachments 1 and 2.)

11.2.7 Add 100 mL 1:1 methylene chloride/acetone to each sample and sonicate for three minutes using a 3/4-inch horn. The power output should be between 60% and 100% with pulse set on 1 second ON and percent duty cycle knob set at 50%. Do <u>not</u> use a microtip. The setting is dependent upon the sample matrix. The analyst MUST observe very active mixing of the sample and solvent and must adjust the output accordingly. Place the bottom of the horn about 1/2 an inch below the surface of the solvent, but above the sediment layer. Rinse the probe end in the beaker with a small amount of the 1:1 mixture. As each sample finishes sonication, decant

into a Buchner funnel containing Munktell 389 filter paper. Use vacuum filtration and collect the extract in a sidearm flask.

11.2.8 Repeat the extraction two more times with additional 100 mL portions of 1:1 methylene chloride: acetone. Before each extraction, thoroughly mix the solid residue, and make certain that the sodium sulfate is free flowing and not a consolidated mass. As needed, break up large lumps with a clean spatula. Decant and filter the extraction solvent after each sonication as above. After the final sonication, pour the entire sample into the funnel and rinse the teflon beaker and funnel with 20 to 30 mL of methylene chloride

11.2.9 After completion of the third extraction, pour the entire sample into the Buchner funnel and rinse with the extraction solvent mixture.

If particulate matter is observed, filter the entire extracted sample again through newly prepared filter paper using the original funnel or filter at a concentrated volume with a .45 µm membrane filter.

NOTE: Do NOT allow the sample to completely dry. Continued application of the vacuum pump may result in the loss of some analytes.

11.3 Extract Concentration

11.3.1 For each sample being concentrated, rinse a Snyder column with methylene chloride.

11.3.2 Pour the extract into a KD apparatus. Add 2-3 boiling chips to each K-D flask and attach a Snyder column. If the Snyder column is not still wet from the rinsing process, add 1-2 mL methylene chloride to the top of the column.

Note: For Method 3550C, the extract is required to be poured through a powder funnel inserted into the top of the KD apparatus and containing a glass wool plug and 10 g of furnaced sodium sulfate.

11.3.3 Place the K-D apparatus on a water bath set at 80-90 °C. The apparatus should be placed in the bath so that the entire lower rounded surface of the flask is bathed in hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10-

15 minutes. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of the sample reaches 4 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

11.3.4 Final concentration volume of semivolatile fractions

11.3.4.1 Remove the Snyder column and K-D, then concentrate the extract to 1.0 mL volume using the nitrogen blowdown technique or microsnyder concentration. Transfer the entire 1.0 mL into a 2 mL amber autosampler vial labeled with the sample number, method (8270, and extraction date. Note the final volume on the Extractions Preparation Bench Sheet.

11.3.4.2 Deliver the Extractions Preparation Bench Sheet and extracts to the semivolatile laboratory and complete chain-of-custody documentation.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4 "Numerical Data Reduction".

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analysts. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96, Methods 3550B and 8270C; Update IV, Methods 3550C and 8270D, 02/07

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Editions upon promulgation, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 NELAC Standards, effective July 2003, plus revisions

16.7 QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April, 2007.

16.8 CompuChem Quality Manual, Revision 14, January 25, 2011, plus revisions

16.9 Sample Control SOP 4.6, "Storing Samples"

16.10 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010, plus revisions

16.11 Sample Control SOP 4.6, "Storing Samples"

16.12 Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

16.13 Instrument Procedure 477, "GC/MS Analysis of Extractable Semivolatiles in Aqueous and Solid Samples by SW-846"

16.14 Organic Extractions Lab SOP 8.2, "Sonication Procedure"

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 – Example Full Scan Spiking Solution Standards Worksheets

17.2 Attachment 2 - Example SIM Spiking Solution Standards Worksheet

17.3 Attachment 3 - Example Extraction Preparation Bench Sheets

Attachment 1

Analytical Standard Record
COMPUCHEM
0I02003

Description:	#393 8270 BNA Surrogate	Expires:	07/31/201
Standard Type:	Surrogate Spike	Prepared:	09/02/201
Solvent:	Methanol	Prepared By:	** Vendor **
Final Volume (mls):	25	Department:	SVOA
Vials:	8	Last Edit:	09/14/2010 13:43 by jer

WL-371-C 073010-39

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	98904-43-9	100	ug/mL
Phenol-d5	4165-62-2	200	ug/mL
Nitrobenzene-d5	4165-60-0	100	ug/mL
2-Fluorophenol	367-12-4	200	ug/mL
2-Fluorobiphenyl	321-60-8	100	ug/mL
2,4,6-Tribromophenol	118-79-6	200	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0J11002

Description:	8270 Validation I	Expires:	04/11/201
Standard Type:	Analyte Spike	Prepared:	10/11/201
Solvent:	MeOH/DB781	Prepared By:	Jaime Redig
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	11/24/2010 11:01 by KG

64480 and co-elution factor

Analyte	CAS Number	Concentration	Units
4-Chloro-3-methylphenol	59-50-7	80	ug/mL
2-Methylnaphthalene	91-57-6	80	ug/mL
2-Methylphenol	95-48-7	80	ug/mL
2-Nitroaniline	88-74-4	80	ug/mL
2-Nitrophenol	88-75-5	80	ug/mL
3 & 4-Methylphenol	106-44-5	80	ug/mL
3-Methylphenol	108-39-4	80	ug/mL
3-Nitroaniline	99-09-2	80	ug/mL
Aniline	62-53-3	80	ug/mL
4-Bromophenyl-phenylether	101-55-3	80	ug/mL
2,6-Dinitrotoluene	606-20-2	80	ug/mL
4-Chloroaniline	106-47-8	80	ug/mL
4-Chlorophenyl-phenylether	7005-72-3	80	ug/mL
4-Methylphenol	106-44-5	80	ug/mL
4-Nitroaniline	100-01-6	80	ug/mL
4-Nitrophenol	100-02-7	80	ug/mL
Acenaphthene	83-32-9	80	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	80	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	80	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	80	ug/mL
1,2,4-Trichlorobenzene	120-82-1	80	ug/mL
1,2-Dichlorobenzene	95-50-1	80	ug/mL
1,2-Dinitrobenzene	528-29-0	80	ug/mL
1,3-Dichlorobenzene	541-73-1	80	ug/mL
1,3-Dinitrobenzene	99-65-0	80	ug/mL
1,4-Dichlorobenzene	106-46-7	80	ug/mL
1,4-Dinitrobenzene	100-25-4	80	ug/mL
2-Chlorophenol	95-57-8	80	ug/mL
2,2'-oxybis(1-Chloropropane)	108-60-1	80	ug/mL
2-Chloronaphthalene	91-58-7	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0J11002

2,3,5,6-Tetrachlorophenol	935-95-5	80	ug/mL
2,4,5-Trichlorophenol	95-95-4	80	ug/mL
2,4,6-Trichlorophenol	88-06-2	80	ug/mL
2,4-Dichlorophenol	120-83-2	80	ug/mL
2,4-Dimethylphenol	105-67-9	80	ug/mL
2,4-Dinitrophenol	51-28-5	80	ug/mL
2,4-Dinitrotoluene	121-14-2	80	ug/mL
Anthracene	120-12-7	80	ug/mL
1-Methylnaphthalene	90-12-0	80	ug/mL
N-Nitrosodimethylamine	62-75-9	80	ug/mL
Fluoranthene	206-44-0	80	ug/mL
Fluorene	86-73-7	80	ug/mL
Hexachlorobenzene	118-74-1	80	ug/mL
Hexachlorobutadiene	87-68-3	80	ug/mL
Hexachlorocyclopentadiene	77-47-4	80	ug/mL
Hexachloroethane	67-72-1	80	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	80	ug/mL
Acenaphthylene	208-96-8	80	ug/mL
N-Nitroso-di-N-propylamine	621-64-7	80	ug/mL
Dibenzofuran	132-64-9	80	ug/mL
N-Nitrosodiphenylamine (1)	86-30-6	80	ug/mL
Naphthalene	91-20-3	80	ug/mL
Nitrobenzene	98-95-3	80	ug/mL
Pentachlorophenol	87-86-5	80	ug/mL
Phenanthrene	85-01-8	80	ug/mL
Phenol	108-95-2	80	ug/mL
Pyrene	129-00-0	80	ug/mL
Isophorone	78-59-1	80	ug/mL
bis(2-ethylhexyl)Phthalate	117-81-7	80	ug/mL
Azobenzene	103-33-3	80	ug/mL
Benzo (a) anthracene	56-55-3	80	ug/mL
Benzo (a) pyrene	50-32-8	80	ug/mL
Benzo (b) fluoranthene	205-99-2	80	ug/mL
Benzo (g,h,i) perylene	191-24-2	80	ug/mL
Benzo(k)fluoranthene	207-08-9	80	ug/mL
Benzyl alcohol	100-51-6	80	ug/mL
Dimethylphthalate	131-11-3	80	ug/mL
Bis(2-chloroethyl)ether	111-44-4	80	ug/mL
Diethylphthalate	84-66-2	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0J11002

Bis-(2-Ethylhexyl) Adipate	103-23-1	80	ug/mL
Butylbenzylphthalate	85-68-7	80	ug/mL
Carbazole	86-74-8	80	ug/mL
Chrysene	218-01-9	80	ug/mL
Di-n-butylphthalate	84-74-2	80	ug/mL
Di-n-octylphthalate	117-84-0	80	ug/mL
Dibenzo(a,h)anthracene	53-70-3	80	ug/mL
Pyridine	110-86-1	80	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	80	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0B17001	NSI C-701 8270 BNA MIX	02/16/201	** Vendor **	01/31/201	07/21/2010 12:16 by EB	2
0C25012	Accustandard 1,2,4,5-Tetrachlorob	03/25/201	** Vendor **	01/29/202	06/24/2010 11:14 by jer	2
0F30010	3 & 4-Methylphenol CO-ELUTION	06/30/201	Jaime Redig	04/04/201	10/29/2010 12:56 by jer	1
0K24005	3 & 4-Methylphenol	11/24/201	Ken Grzybowski	11/19/201	11/24/2010 10:59 by KG	2

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0J11003

Description:	8270 Validation II Spike	Expires:	04/11/201
Standard Type:	Analyte Spike	Prepared:	10/11/201
Solvent:	MeOH/DB781	Prepared By:	Jaime Redig
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	11/29/2010 07:25 by jer

64481

Analyte	CAS Number	Concentration	Units
Caprolactam	105-60-2	80	ug/mL
Benzidine	92-87-5	80	ug/mL
Benzaldehyde	100-52-7	80	ug/mL
Atrazine	1912-24-9	80	ug/mL
Acetophenone	98-86-2	80	ug/mL
3,3'-Dimethylbenzidine	119-93-7	80	ug/mL
3,3'-Dichlorobenzidine	91-94-1	80	ug/mL
1,1'-Biphenyl	92-52-4	80	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0H05002	Absolute OLMO4.1 Additionals	08/05/201	** Vendor **	07/01/201	11/29/2010 07:25 by jer	1
0J01011	Restek@31852 8270 Benzidines M	10/01/201	** Vendor **	05/31/201	10/01/2010 17:02 by jer	1

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Analytical Standard Record
COMPUCHEM
0G23002

Description:	APP IX Spike I	Expires:	01/23/201
Standard Type:	Analyte Spike	Prepared:	07/23/201
Solvent:	Methanol/48115	Prepared By:	Jaime Redig
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	11/01/2010 08:43 by jer

64226

Analyte	CAS Number	Concentration	Units
4-Aminobiphenyl	92-67-1	80	ug/mL
Dimethoate	60-51-5	80	ug/mL
Dibenz (a,j) acridine	224-42-0	80	ug/mL
Diallate	2303-16-4	80	ug/mL
Chlorobenzilate	510-15-6	80	ug/mL
Caprolactam	105-60-2	80	ug/mL
Benzidine	92-87-5	80	ug/mL
Benzaldehyde	100-52-7	80	ug/mL
Atrazine	1912-24-9	80	ug/mL
Aramite	140-57-8	80	ug/mL
Acetophenone	98-86-2	80	ug/mL
a,a-Dimethylphenethylamine	122-09-8	80	ug/mL
7,12-Dimethylbenz(a)anthracene	57-97-6	80	ug/mL
1,1'-Biphenyl	92-52-4	80	ug/mL
4-Nitroquinoline-1-oxide	56-57-5	80	ug/mL
Ethyl Methacrylate	97-63-2	80	ug/mL
3-Methylcholanthrene	56-49-5	80	ug/mL
3,3'-Dimethylbenzidine	119-93-7	160	ug/mL
3,3'-Dichlorobenzidine	91-94-1	80	ug/mL
2-Picoline	109-06-8	80	ug/mL
2-Naphthylamine	91-59-8	80	ug/mL
2-Acetylaminofluorene	53-96-3	80	ug/mL
2,6-Dichlorophenol	87-65-0	80	ug/mL
1-Naphthylamine	134-32-7	80	ug/mL
1-Chloronaphthalene	90-13-1	80	ug/mL
1,4-Naphthoquinone	130-15-4	80	ug/mL
1,4-Dioxane	123-91-1	80	ug/mL
1,3,5-Trinitrobenzene	99-35-4	80	ug/mL
1,2,4,5-Tetrachlorobenzene	95-94-3	80	ug/mL
5-Nitro-o-toluidine	99-55-8	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0G23002

N-Nitrosomorpholine	59-89-2	80	ug/mL
Sulfotepp	3689-24-5	80	ug/mL
Safrole	94-59-7	80	ug/mL
Pronamide	23950-58-5	80	ug/mL
Phorate	298-02-2	80	ug/mL
Phenacetin	62-44-2	80	ug/mL
Pentachloronitrobenzene	82-68-8	80	ug/mL
Pentachloroethane	76-01-7	80	ug/mL
Pentachlorobenzene	608-93-5	80	ug/mL
Parathion	56-38-2	80	ug/mL
p-Phenylenediamine	106-50-3	80	ug/mL
p-Dimethylaminoazobenzene	60-11-7	80	ug/mL
o-Toluidine	95-53-4	80	ug/mL
o,o,o-Triethyl phosphorothioate	126-68-1	80	ug/mL
Diphenyl ether	101-84-8	80	ug/mL
Methapyrilene	91-80-5	80	ug/mL
Zinophos	297-97-2	80	ug/mL
Ethyl Methanesulfonate	62-50-0	80	ug/mL
Famphur	52-85-7	80	ug/mL
Hexachloropropene	1888-71-7	80	ug/mL
Isodrin	465-73-6	80	ug/mL
N-Nitrosopyrrolidine	930-55-2	80	ug/mL
Kepone	143-50-0	80	ug/mL
N-Nitrosopiperidine	100-75-4	80	ug/mL
Methyl Methanesulfonate	66-27-3	80	ug/mL
Methyl Parathion	298-00-0	80	ug/mL
N-Nitrosodibutylamine	924-16-3	80	ug/mL
N-Nitrosodiethylamine	55-18-5	80	ug/mL
N-Nitrosomethylethylamine	10595-95-6	80	ug/mL
Disulfoton	298-04-4	80	ug/mL
Isosafrole	120-58-1	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0G23002

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0C03011	Restek@31625 APP IX Mix 1	03/03/201	** Vendor **	03/31/201	07/02/2010 12:17 by jer	1
0C03012	Restek@32419 8270 OP Pest Mix	03/03/201	** Vendor **	03/31/201	05/14/2010 13:20 by jer	1
0D21008	Restek@31852 8270 Benzidines M	04/21/201	** Vendor **	03/31/201	07/21/2010 11:17 by RKM	1
0E24009	Restek@563626 APP IX Mix 2	05/24/201	** Vendor **	07/31/201	08/04/2010 14:04 by jer	2
9J28005	NSI 1,3,5 TRINITROBENZENE	10/28/200	Rodney Mckoy	09/30/201	05/14/2010 13:21 by jer	2

Reviewed By _____ Date _____

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0G23003

Description:	APP IX Spike II	Expires:	01/23/201
Standard Type:	Analyte Spike	Prepared:	07/23/201
Solvent:	Methanol/48115	Prepared By:	Jaime Redig
Final Volume (mls):	25	Department:	SVOA
Vials:	1	Last Edit:	11/01/2010 08:43 by jer

64227

Analyte	CAS Number	Concentration	Units
4-Chloroaniline	106-47-8	80	ug/mL
Anthracene	120-12-7	80	ug/mL
2-Methylphenol	95-48-7	80	ug/mL
2-Nitroaniline	88-74-4	80	ug/mL
2-Nitrophenol	88-75-5	80	ug/mL
3-Methylphenol	108-39-4	80	ug/mL
3-Nitroaniline	99-09-2	80	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	80	ug/mL
2-Chlorophenol	95-57-8	80	ug/mL
4-Chloro-3-methylphenol	59-50-7	80	ug/mL
2-Chloronaphthalene	91-58-7	80	ug/mL
4-Chlorophenyl-phenylether	7005-72-3	80	ug/mL
4-Methylphenol	106-44-5	80	ug/mL
4-Nitroaniline	100-01-6	80	ug/mL
4-Nitrophenol	100-02-7	80	ug/mL
Acenaphthene	83-32-9	80	ug/mL
Acenaphthylene	208-96-8	80	ug/mL
1,2,4-Trichlorobenzene	120-82-1	80	ug/mL
4-Bromophenyl-phenylether	101-55-3	80	ug/mL
2,3,5,6-Tetrachlorophenol	935-95-5	80	ug/mL
1,2-Dichlorobenzene	95-50-1	80	ug/mL
1,2-Dinitrobenzene	528-29-0	80	ug/mL
1,3-Dichlorobenzene	541-73-1	80	ug/mL
1,3-Dinitrobenzene	99-65-0	80	ug/mL
1,4-Dichlorobenzene	106-46-7	80	ug/mL
1,4-Dinitrobenzene	100-25-4	80	ug/mL
1-Methylnaphthalene	90-12-0	80	ug/mL
2-Methylnaphthalene	91-57-6	80	ug/mL
2,3,4,6-Tetrachlorophenol	58-90-2	80	ug/mL
Azobenzene	103-33-3	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0G23003

2,4,5-Trichlorophenol	95-95-4	80	ug/mL
2,4,6-Trichlorophenol	88-06-2	80	ug/mL
2,4-Dichlorophenol	120-83-2	80	ug/mL
2,4-Dimethylphenol	105-67-9	80	ug/mL
2,4-Dinitrophenol	51-28-5	80	ug/mL
2,4-Dinitrotoluene	121-14-2	80	ug/mL
2,6-Dinitrotoluene	606-20-2	80	ug/mL
2,2'-oxybis(1-Chloropropane)	108-60-1	80	ug/mL
N-Nitrosodimethylamine	62-75-9	80	ug/mL
Aniline	62-53-3	80	ug/mL
Fluorene	86-73-7	80	ug/mL
Hexachlorobenzene	118-74-1	80	ug/mL
Hexachlorobutadiene	87-68-3	80	ug/mL
Hexachlorocyclopentadiene	77-47-4	80	ug/mL
Hexachloroethane	67-72-1	80	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	80	ug/mL
Dimethylphthalate	131-11-3	80	ug/mL
N-Nitroso-di-N-propylamine	621-64-7	80	ug/mL
Diethylphthalate	84-66-2	80	ug/mL
N-Nitrosodiphenylamine (1)	86-30-6	80	ug/mL
Naphthalene	91-20-3	80	ug/mL
Nitrobenzene	98-95-3	80	ug/mL
Pentachlorophenol	87-86-5	80	ug/mL
Phenanthrene	85-01-8	80	ug/mL
Phenol	108-95-2	80	ug/mL
Pyrene	129-00-0	80	ug/mL
Isophorone	78-59-1	80	ug/mL
Bis-(2-Ethylhexyl) Adipate	103-23-1	80	ug/mL
Benzo (a) anthracene	56-55-3	80	ug/mL
Benzo (a) pyrene	50-32-8	80	ug/mL
Benzo (b) fluoranthene	205-99-2	80	ug/mL
Benzo (g,h,i) perylene	191-24-2	80	ug/mL
Benzo(k)fluoranthene	207-08-9	80	ug/mL
Benzyl alcohol	100-51-6	80	ug/mL
Bis(2-chloroethoxy)methane	111-91-1	80	ug/mL
Fluoranthene	206-44-0	80	ug/mL
bis(2-ethylhexyl)Phthalate	117-81-7	80	ug/mL
Pyridine	110-86-1	80	ug/mL
Butylbenzylphthalate	85-68-7	80	ug/mL

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Attachment 1 (Continued)

Analytical Standard Record
COMPUCHEM
0G23003

Carbazole	86-74-8	80	ug/mL
Chrysene	218-01-9	80	ug/mL
Di-n-butylphthalate	84-74-2	80	ug/mL
Di-n-octylphthalate	117-84-0	80	ug/mL
Dibenzo(a,h)anthracene	53-70-3	80	ug/mL
Dibenzofuran	132-64-9	80	ug/mL
Bis(2-chloroethyl)ether	111-44-4	80	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0B17001	NSI C-701 8270 BNA MIX	02/16/201	** Vendor **	01/31/201	07/21/2010 12:16 by EB	2
0F30010	3 & 4-Methylphenol CO-ELUTION	06/30/201	Jaime Redig	04/04/201	10/29/2010 12:56 by jer	1

Reviewed By _____ Date _____

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Attachment 2

Analytical Standard Record
COMPUCHEM
0J11001

Description:	Dil 393	Expires:	04/11/201
Standard Type:	Surrogate Spike	Prepared:	10/11/201
Solvent:	MeOH/DB781	Prepared By:	Jaime Redig
Final Volume (mls):	100	Department:	SVOA
Vials:	1	Last Edit:	10/14/2010 10:08 by jer

64479

Analyte	CAS Number	Concentration	Units
Terphenyl-d14	98904-43-9	1	ug/mL
Phenol-d5	4165-62-2	2	ug/mL
Nitrobenzene-d5	4165-60-0	1	ug/mL
2-Fluorophenol	367-12-4	2	ug/mL
2-Fluorobiphenyl	321-60-8	1	ug/mL
2,4,6-Tribromophenol	118-79-6	2	ug/mL

Parent Standards used in this standard:

Standard	Description	Prepared	Prepared By	Expires	Last Edit	(mls)
0I02003	#393 8270 BNA Surrogate	09/02/201	** Vendor **	07/31/201	09/14/2010 13:43 by jer	1

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Attachment 2 (Continued)

Analytical Standard Record
COMPUCHEM
1E04002

Description:	NSI SIM Spike Q-6605-O	Expires:	02/28/2013
Standard Type:	Analyte Spike	Prepared:	05/04/2011
Solvent:	MeOH	Prepared By:	Brad Lake
Final Volume (mls):	25	Department:	SVOA
Vials:	5	Last Edit:	05/13/2011 15:35 by BWL

021011-01

Analyte	CAS Number	Concentration	Units
Chrysene	218-01-9	1	ug/mL
2,4-Dinitrophenol	51-28-5	4	ug/mL
2-Methylnaphthalene	91-57-6	1	ug/mL
4,6-Dinitro-2-methylphenol	534-52-1	4	ug/mL
4-Nitrophenol	100-02-7	4	ug/mL
Acenaphthene	83-32-9	1	ug/mL
Acenaphthylene	208-96-8	1	ug/mL
Anthracene	120-12-7	1	ug/mL
Benzo (a) anthracene	56-55-3	1	ug/mL
Benzo (a) pyrene	50-32-8	1	ug/mL
Benzo (b) fluoranthene	205-99-2	1	ug/mL
1-Methylnaphthalene	90-12-0	1	ug/mL
Benzo(k)fluoranthene	207-08-9	1	ug/mL
Pyrene	129-00-0	1	ug/mL
Dibenzo(a,h)anthracene	53-70-3	1	ug/mL
Dibenzofuran	132-64-9	1	ug/mL
Fluoranthene	206-44-0	1	ug/mL
Fluorene	86-73-7	1	ug/mL
Hexachlorobenzene	118-74-1	4	ug/mL
Hexachlorobutadiene	87-68-3	1	ug/mL
Indeno(1,2,3-cd)pyrene	193-39-5	1	ug/mL
Naphthalene	91-20-3	1	ug/mL
Pentachlorophenol	87-86-5	4	ug/mL
Phenanthrene	85-01-8	1	ug/mL
Benzo (g,h,i) perylene	191-24-2	1	ug/mL

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Attachment 3

Assigned To _____

Employee ID Number _____

Matrix: Soil

PREPARATION BENCH SHEET

1053011

SVOC 8270D

Prepared using: SVOA - 3550B/C

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial (g)	Final (uL)	QC	Surr (uL)	Comments
1053011-BLK1	SBLKMY	Blank				500	
1053011-BS1	SMYLCS	LCS				500	
1053011-BSD1	SMYLCSD	LCS Dup				500	
1053011-MS1	SB-1-1-2MS	Matrix Spike				500	
1053011-MSD1	SB-1-1-2MSD	Matrix Spike Dup				500	
1105172-01	SB-1-1-2	Sample				500	
1105172-02	SB-1-15-16	Sample				500	
1105172-03	SB-2-1-2	Sample				500	
1105172-04	SB-2-15-16	Sample				500	
1105172-05	SB-3-0-1	Sample				500	
1105172-06	SB-3-15-16	Sample				500	
1105172-07	SB-4-0-1	Sample				500	
1105172-08	SB-4-15-16	Sample				500	
1105172-09	SB-5-0-1	Sample				500	
1105172-10	SB-5-15-16	Sample				500	
1105172-11	SB-6-0-1	Sample				500	
1105172-12	SB-6-3-4	Sample				500	
1105172-13	SB-7-0-1	Sample				500	
1105172-14	SB-7-3-4	Sample				500	
1105172-15	SB-8-0-1	Sample				500	
1105172-16	SB-8-3-4	Sample				500	

	Description	Spike Amount (uL)	Lot Number
SURROGATE	#393 8270 BNA Surrogate	500	
SPIKE	8270 Validation I	500	LCS/LCSD
SPIKE	8270 Validation I	500	MS/MSD
SPIKE	8270 Validation II Spike	500	LCS/LCSD
SPIKE	8270 Validation II Spike	500	MS/MSD

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

Balance ID: _____

Manuf. and lot # of reagents/solvents used: _____

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Attachment 3 (Continued)

Assigned To _____

Employee ID Number _____

Matrix: Soil

PREPARATION BENCH SHEET

1052515

SVOC 8270D SIM FULL DOD

Prepared using: SVOA - 3550B/C

Date/Time Extracted: _____

Lab Number	Client ID	QCType	Initial (g)	Final (uL)	QC	Surr (uL)	Comments
1052515-BLK1	SBLKLP	Blank				500	
1052515-BS1	SLPLCS	LCS				500	
1052515-MS1	74SB535-01MS	Matrix Spike				500	
1052515-MSD1	74SB535-01MSD	Matrix Spike Dup				500	
1105118-16	74SB526-00	Sample				500	
1105118-17	74SB526-00D	Sample				500	
1105118-18	74SB534-00	Sample				500	
1105118-19	74SB535-00	Sample				500	
1105118-20	74SB541-00	Sample				500	
1105130-12	74SB678G-00	Sample				500	
1105130-13	74SB678G-01	Sample				500	
1105130-14	74SB678G-05	Sample				500	
1105130-15	74SB678G-07	Sample				500	
1105140-20	74SB535-01	Sample			QC	500	

	Description	Spike Amount (uL)		Lot Number
SURROGATE	Dil 393	500		
SPIKE	NSI SIM Spike Q-6605-O	500	LCS/LCSD	
SPIKE	NSI SIM Spike Q-6605-O	500	MS/MSD	

Surrogate & Spike Added By: _____
Initials / Date

Spiking Witnessed By: _____
Initials / Date

Final Vol Verified: _____

Reviewed By: _____

Analysts Initials: Extracted: _____ KD: _____ N2: _____ Bottled up: _____

Balance ID: _____

Manuf. and lot # of reagents/solvents used: _____

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP - 814 SOP Section #: 27.2 Revision #: 11

SOP Title: Toxicity Characteristic Leaching Procedure (TCLP) by SW-846 Method 1311

Effective date: (QA fills in) 10-30-2009

- Procedure prepared by: [signature] Date: 10-28-2009
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 10-29-09
- Reason for change: Annual Review, SC DHEC Compliance
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Ed. Update I, 7/92

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: [signature] Date: 3-8-10

Annual Review—Signature: Bob Meier Date: 06-14-2011

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -814: Toxicity Characteristic Leaching Procedure (TCLP) by SW-846 **Method 1311**

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Sample Preparation Procedure -814: Toxicity Characteristic Leaching Procedure (TCLP) by SW-846 **Method 1311**

1.0 Scope and Application

The TCLP is designed to determine the mobility of both organic and inorganic contaminants present in liquid, solid, and multiphasic waste. Constituents and regulatory levels are found in Attachment 1. **All data being reported to SC DHEC must adhere to permit specified limits.**

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste after filtration through a 0.6- to 0.8-µm glass fiber filter is defined as the TCLP extract. For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the solid phase, if necessary, is reduced in particle size and is followed by leaching.

The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. After the extraction, the liquid extract is separated from the solid phases by filtration through a 0.6- to 0.8-µm glass fiber filter. If compatible (multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately, and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 Definition

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)

3.2 Reporting Limit – The laboratory reporting limit is based on the lowest multipoint calibration standard concentration. **All data being reported to SC DHEC must adhere to permit specified limits.**

For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

For some inorganic methods, the reporting limit is based on the MDL, and is usually 4-8 times higher than the MDL. This is referred to as the Practical Quantitation Limit (PQL). Values detected between the PQL and the instrument detection limit (IDL) are flagged and reported.

3.3 Reporting Units – ug/L

3.4 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

Note: The **DoD-QSM** and SC Department of Health and Environmental Control (SC DHEC) do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5 **% for DoD-QSM** and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.5 DoD-QSM – Department of Defense Quality Systems Manual

3.6 SC DHEC – South Carolina Department of Health and Environmental Control

4.0 Interferences

4.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods and SOPs.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory **staff members** are **required** to review the Chemical Hygiene Plan for general safety **policies** and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment & Supplies

6.1 Agitation apparatus

6.1.1 A rotating extraction vessel in an end-over-end fashion at 30 ± 2 rpm as diagrammed in Attachment 2.

6.2 Bottle extract vessel

6.2.1 For nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed.

6.2.2 Headspace is allowed in the vessel.

6.2.3 It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern.

6.2.4 Plastic bottles other than polytetrafluoroethylene shall not be used if organics are to be investigated.

6.3 Filtration devices

Note: It is recommended that all filtrations be performed in a hood.

When the waste is evaluated for anything except volatile compounds, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation (50 psi) may be used.

6.4 Filters (Attachment 3 contains additional information on filters and filter holders.)

6.4.1 Filters are made of borosilicate glass fiber, do not contain binder materials, and have an effective pore size of 0.6- to 0.8- µm or equivalent.

6.4.2.1 Do not use prefilters.

6.4.2 Whatman Lab. Products, Inc., Filters G,F/F, pore size 0.7 µm

6.5 Balance

6.5.1 Any balance accurate to within 0.01 g may be used.

6.6 pH meter--accuracy of ± 0.05 units at 25°C

6.7 500-**mL** glass beakers

6.8 Watch glasses

6.9 Magnetic stirrer

7.0 Reagents & Standards

Standards are stored separately from samples at 4° C ± 2° C in the laboratory when not in use.

Refer to the Standards Preparation Logbook 22 F or the Promium Element LIMs for details on preparation of standards used in this procedure.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

7.1 Reagent water – All water used in this procedure must be equivalent to ASTM Type II water (as it relates to specific conductance and specific resistance) which is demonstrated to meet the blank contamination acceptance criteria contained in this Standard Operating Procedure (SOP). It is referred to throughout the remainder of this SOP as DI water.

7.2 1.0 N Hydrochloric acid (HCL) - ACS reagent grade, concentrated

7.2.1 To prepare dilute 20 **mL** of a 1:1 solution of concentrated HCL to a volume of 1000 m**L** with DI water.

7.3 1.0 N nitric acid (HNO_3)

7.3.1 Dilute 63 **mL of concentrated HNO_3 - ACS reagent grade** to 1000 **mL** with DI water.

7.4 1.0 N sodium hydroxide (NaOH)

7.4.1 Dilute 40 g **of NaOH - ACS reagent grade** to 1000 **mL** with DI water.

7.5 Glacial acetic acid (HOAc) - ACS reagent grade, concentrated

7.6 Extraction fluid:

7.6.1 Extraction fluid #1--This fluid is made by adding 5.7 **mL** glacial HOAc to 500 **mL** of **DI water**, adding 60 **mL** of 1.0 N NaOH, and diluting **with DI water** to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05. **To** make 6 liters **(a typical volume prepared) of extraction fluid #1: add** 34.2 **mL** glacial HOAc to 3000 **mL** DI water**, t**hen add 360 **mL** 1.0N NaOH and dilute to 6 **l**iters **with DI water**.

7.6.2 Extraction fluid #2--This fluid is made by diluting 5.7 **mL** glacial HOAc with **DI water** to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05. **To make 6 liters (a typical volume prepared) of extraction fluid #2:** 34.2 **mL** glacial HOAc **is** diluted to 6 liters with DI water.

Note: Extraction fluids are made fresh daily. The pH must be checked prior to use to ensure accuracy. Record the pH on the TCLP Extraction Worksheet (Attachment 4) for the blanks.

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 All extracts must be analyzed within 40 days of extraction. (HPLC, GC and GCMS non-volatiles.)

8.3 Prior to analysis, all extracts must be stored under refrigeration at **4° C ± 2° C** in the reach-in storage unit in the laboratory. After analysis, extracts are returned to Sample Control for long-term storage and disposal. (HPLC, GC and GCMS non-volatiles.)

8.4 Samples are obtained from the **Sample** Custodian out of cold storage. They **must** be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the **Sample** Custodian and placed in the cooler at 4° C ± **2° C** for long-term storage and disposal.

8.5 Preservatives are not added to samples prior to extraction.

8.6 Samples can be refrigerated unless it results in irreversible physical changes to the waste.

8.7 TCLP extracts should be prepared for analysis and analyzed as soon as possible after extraction. If they need to be stored, even for a short time, storage shall be at 4°C ± **2° C**.

Sample Maximum Holding Times (Days)

From:	Field Collection	1311 Extraction	Preparative Extraction	Total Elapsed Time
To:	1311 Extraction	Preparative Extraction	Analysis	
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Metals	180	NA	180	360
Mercury	28	NA	28	56

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 Quality Control

9.1 A leachate blank is prepared with each sample batch. The leachate blank must meet the method specific preparative extraction and analysis SOP requirements.

9.2 All other quality control objectives must be meet as detailed in each method specific preparative extraction and analysis SOP.

10.0 Calibration & Standardization

10.1 The pH meter must be calibrated on each day of use and must be recorded in the corresponding pH meter Calibration Logbook (attachment 5). Include the lot number of the buffer solution being used.

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11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures.

11.1 Procedure Used when Volatiles are not involved

Although a minimum sample size of 100 g is required, a larger sample size may be necessary, depending on the percent solids of the waste sample. Enough **of each** waste sample **must** be collected so that at least 75 g of the solid phase of the waste (as determined using glass fiber filter filtration) is extracted. This will ensure that there is adequate extract for the required analyses (e.g., semivolatiles, metals, pesticides, and herbicides).

Sections 11.1 through 11.4 are the necessary preliminary evaluations. Perform preliminary evaluations on a minimum of a 100-g aliquot of waste.

11.1.1 Percent Solids Determination

11.1.1.1 If the waste will obviously yield no free liquid when subjected to pressure filtration, weigh out a representative subsample of the waste (100 g minimum) and proceed to section 11.1.3 for particle-size reduction.

11.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation is required to determine percent solids. This is performed using a Millipore filtration device.

11.1.1.3 Pre-weigh the filter and the container which will receive the filtrate. Record **the weights** on the worksheet.

11.1.1.4 Following the manufacturer's instructions assemble the filter holder and filter. Place the filter on the support screen and secure.

11.1.1.5 Mix thoroughly to achieve homogeneity. Discard any foreign objects such as sticks, stones, and

leaves. Pre-weigh the container and record the weight on the worksheet. Weigh out a representative subsample of the waste (100-g minimum) and record the weight.

11.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged before filtration.

11.1.1.7 Transfer the waste sample to the filter holder.

Note: If waste material has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in step 11.1.1.5, to determine the weight of the waste sample that will be filtered.

When the pressurizing gas begins to move through, **gradually** apply a vacuum or gentle pressure of 1-10 psi until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2-minute interval, slowly increase the pressure in 10-psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter and if no additional liquid has passed through the filter in any 2-minute interval, proceed to the next the filter or when liquid flow has ceased at 50 psi (i.e., does not result in any additional filtrate within any 2-minute period), filtration is stopped.

Note: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

11.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

Note: Some wastes, such as oil wastes and some paint wastes, **may** contain some material which **while** appear**ing** to be a liquid**, will not filter, even** after applying vacuum or pressure filtration, as outlined in step 11.1.1.7**. When** this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid.

11.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see step 11.1.1.3). The weight of the solid phase of the waste sample is determined by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in step 11.1.1.5 or 11.1.1.7. See 12.9.5 for the calculation. Record the weight of the liquid and solid phases.

11.2 Determine whether the waste contains insignificant solids, and is, therefore, its own TCLP extract after filtration.

11.2.1 The sample will be handled differently from this point, depending on whether it contains more or less than 0.5% dry solids. If the sample obviously has greater than 0.5% dry solids go to step 11.3. If it appears that the solid may comprise less than 0.5% of the total waste, the percent dry solids will be determined as follows.

11.2.2 Remove the solid phase and filter from the filtration apparatus.

11.2.3 Dry the filter and solid phase at 100 ± 20° C until two successive weighings yield the same value. Record **the** final weight. Calculate the percent dry solids using the calculation in 12.9.6.

11.2.4 If the solid comprises less than 0.5% of the dried waste, the solid is discarded and the liquid phase is defined as the TCLP extract. A filter blank must also be **performed**.

11.2.5 If the solid is greater than or equal to 0.5% of the waste, proceed to 11.3 for particle-size reduction.

Note: The dried solid derived from this step is not subjected to extraction. A new sample will have to be prepared for extraction.

11.3 Determination of whether the solid portion of the waste requires particle-size reduction

11.3.1 If the sample has more than 0.5% solids, it is now evaluated for particle size. If the solid material has a surface area per gram of material equal to or greater than 3.1 cm^2, or is capable of passing through a 9.5-mm (0.375-inch) standard sieve, proceed to step 6.4.

11.3.2 If the surface area is smaller or the particle size is larger than that described above, the solid material needs to undergo particle-size reduction.

11.3.3 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a size capable of passing through a 9.5-mm standard sieve.

11.3.4 Use mortar and pestle or other appropriate means (which may include hammer or scissors) to reduce particle size.

11.4 Determination of appropriate extraction fluid

11.4.1 Weigh out a small sub-sample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer a 5.0-g portion to a beaker or Erlenmeyer flask.

11.4.2 Add 96.5 **mL** DI water and stir vigorously for five minutes using a magnetic stirrer. Measure and record the pH. If the pH is < 5.0, extraction fluid #1 is used. Proceed to step 11.5.

11.4.3 If the pH from step 11.4.2 is > 5.0, add 3.5 **mL** 1.0 N HCL, slurry for 30 seconds, heat to 50° C and hold for 10 minutes. Do not stir while heating.

11.4.4 Let the solution cool to room temperature and record the pH. If the pH is < 5.0, use extraction fluid #1. If the pH is greater than or equal to 5.0, use extraction fluid #2.

11.5 Preparation of TCLP extract

11.5.1 A minimum sample size of 100 g (solid and liquid phases) is required. In some cases, a larger sample size may be appropriate depending on the solids content of the waste sample (percent solids); whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid; and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids **must** be generated for extraction such that the volume of TCLP extract will be **sufficient** to support all the analyses required.

11.5.2 Acid wash filters if evaluating the mobility of metals.

Note: Acid-washed filters may be used for all nonvolatile extractions even when metals are not of concern.

To acid-wash filters, rinse filter with 1.0 N nitric acid followed by three consecutive rinses with DI water (a minimum of one liter per rinse is recommended). Alternatively, filters may be purchased already acid washed, and a DI water rinse is done in the laboratory prior to use. Glass fiber filters are fragile and **must** be handled with care.

11.5.3 Filter enough **of each** waste sample to generate 100 g of solids. Save the filtrate in a clean, glass, acid-washed liter bottle. **A plastic bottle is acceptable if the extract is to be analyzed only for inorganics.** Label with CC#, date and filtrate. To calculate how much raw sample is needed, use the equation in 12.9.7.

11.5.4 Quantitatively transfer the solid material into the extraction bottle along with the filter used to separate the initial liquid from the solid phase.

11.5.5 Slowly add the appropriate amount of extraction fluid to the extractor vessel as follows: For every 100 g of solids, add 2000

mL of appropriate extraction fluid (i.e., 20 times the amount of solids used).

11.5.**6** Close the extractor bottle tightly. (It is recommended that Teflon tape be used to ensure a tight seal.) Secure in rotary agitation device and rotate 30 ± 2 rpm for 18 ± 2 hours. To calculate RPM, count the RPM for 30 sec and multiply by 2. Record this number in the designated space on the worksheet.

Note: As agitation continues, pressure may build within the extractor bottle for some types of wastes (e.g., waste containing lime or calcium carbonate may evolve gases such as carbon dioxide). To relieve excess pressure, the extraction bottle may be periodically opened (i.e., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

Ambient temperature must be maintained at 23 ± 2° C during the extraction period. Record the room temperature **on** the TCLP **extraction worksheet** (Attachment **4**) each day of use.

11.5.**7** Samples are not allowed to stand for more than two hours following the 18 ± 2-hour extraction. The material in the extractor vessel is separated into its component liquid and solid phases by pressure filtering through a new glass fiber filter into clean acid-washed 1-liter bottles. This new filter is to be acid-washed if evaluating the mobility of metals unless purchased pre-acid wasted. The glass fiber filter may be changed to facilitate filtration.

11.5.**8** The final pH of the samples is measured and recorded on the worksheet.

11.6 Completing the TCLP extract

11.6.1 If the waste contained no initial liquid phase (filtrate), the filtered liquid material obtained from Step 11.5.**7** is defined as the TCLP extract. Proceed to step 11.7.

11.6.2 If compatible (e.g., will not form precipitate or multiple phases), the filtered liquid resulting from step 11.5.**7** is combined with the initial liquid phase of the waste as obtained in step 11.5.**3**. This

combined liquid is defined as the TCLP extract. Proceed to step 11.7.

11.6.3 If the initial liquid phase of the waste, as obtained from step 11.5.**3**, is not compatible with the filtered liquid resulting from step 11.5.**7**, these liquids are not combined. These liquids are collectively defined as the TCLP extract, are analyzed separately, and the results are combined mathematically. Proceed to step 11.7.

11.7 Label bottles with blue labels as follows:

CompuChem number (CC#)
-814
Date leachate started

If the two phases are not compatible, label filtrate as follows and notify your supervisor immediately since a new CC# will have to be generated for the new fraction.

CompuChem number
-814
Incompatible filtrate
Date

11.8 Make sure the worksheet is filled out correctly.

11.9 If leachates are to be used for metal analysis, notify the inorganics supervisor that the leachates are ready to be subsampled so that the acid preservation step may be performed.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{(Value\ 1\ +\ Value\ 2)/2} x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value\ -\ \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Calibration factors for GC analysis are calculated by dividing the total peak area for each chosen Aroclor component in the standard by the total mass injected (in nanograms).

$$Calibration\ Factor\ (CF) = \frac{Peak\ area}{Total\ mass\ injected}$$

12.8 Calculation of Relative Response Factor (RRF)

$$RRF = \frac{Ax}{Ais} x \frac{Cis}{Cx}$$

where: Ax = area of the characteristic ion from the EICP for the compound to be measured
A_{is} = area of the characteristic ion for the EICP for the internal standard
C_{is} = concentration of the internal standard
C_x = concentration of the compound to be measured

12.9 Concentration

12.9.1 Concentration of aqueous samples for GC analysis

$$ug / L = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Vo)}$$

where: Ax = area response for the analyte
Vt = volume of the concentrated extract (µl)
Df = dilution factor. If no dilution, Df = 1.0
$\overline{CF}$ = mean calibration factor from the initial calibration
Vo = volume of water sample extracted (**mL**)
Vi = volume of extract injected (µl)

12.9.2 Concentration of soil samples <u>(dry weight basis)</u> for GC analysis

$$ug / kg = \frac{(Ax)(Vt)(Df)}{(\overline{CF})(Vi)(Ws)(D)}$$

where: Ws = weight of sample extracted, in grams

D (dry weight) = $\frac{100 - \%\ moisture}{100}$

Ax, Vt, Df, $\overline{CF}$, Vi have the same definitions as for water.

12.9.3 Concentration of aqueous samples by GC/MS analysis

$$ug / L = \frac{(Ax)(Is)(Vt)(Df)}{(Ais)(\overline{RRF})(Vo)(Vi)}$$

where: Ax = area of the characteristic ion from the EICP for the compound to be measured

Ais = area of the characteristic ion for the EICP for the internal standard

Is = amount of internal standard injected (ng)

$\overline{RRF}$ = mean relative response factor from initial calibration standards

Vo = volume of water extracted (**mL**)

Vi = volume of extract injected (µl)

Vt = volume of the concentrated extract (µl)

Df = dilution factor. If no dilution, Df = 1.0

12.9.4 Concentration of soil samples (dry weight basis) by GC/MS

$$ug / kg = \frac{(Ax)(Is)(Vt)(Df)(2.0)}{(Ais)(\overline{RRF})(Vi)(Ws)(D)}$$

where: Ax, Ais, Is, Vt, Vi, RRF, and Df are the same as given for water

2.0 = GPC factor (if used)

Ws = weight of sample extracted, in grams

D (dry weight)= $\frac{100 - \%\ moisture}{100}$

12.9.5 Calculate the percent solids from step 11.1.1.9.

$$Percent\ solids = \frac{weight\ of \cdots solids}{total\ weight\ of\ waste}\ x\ 100$$

12.9.6 Calculate the percent dry solids from steps 11.1.1.5 through 11.1.1.7.

$$percent\ dry\ solids = \frac{weight\ of\ dry\ waste\ and\ filter - weight\ of\ filter}{inital\ weight\ of\ waste}\ x\ 100$$

12.9.7 Calculate how much raw sample is needed from step 11.5.3, use the following equation:

$$To\ obtain\ 100\%\ solids = \frac{100\ grams}{\%\ solids}\ x\ 100$$

12.10 Calculating Dilutions

If a sample concentration exceeds the high level standard a dilution must be performed. Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution. A 10x dilution is performed using 1 **mL** sample plus 9 **mL** diluent for a total volume of 10 mL. It **is** recorded on the run log as "10x (1 mL in 10 mL)."

12.12.1 Dilution factor

$$Dilution\ factor = \frac{\mu L\ most\ conc.\ extract\ used\ to\ make\ dilution + \mu L\ clean\ solvent}{\mu L\ most\ conc.\ extract\ used\ to\ make\ dilution}$$

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. See the method specific preparative extraction and analysis SOPs for this information. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste

generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, H_2SO_4, NaOH, **and ZN** acetate are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW846, 3rd Edition, Update III, 12/96, Method 1311, Revision 0, 7/92

16.2 Federal Register Hazardous Waste Management System, Identification and Testing of Hazardous Waste, Toxicity Characteristic, Final Rule (Vol. 55, No. 61, 6/29/90).

16.3 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.4 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.5 ~~QCSOP:~~ QC SOP 13.6: "Proper Documentation Procedures"

16.6 ~~QCSOP:~~ QC SOP 13.4: "Numerical Data Reduction"

PM 06-14-2011

16.7 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.8 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.9 NELAC Standards, **effective July** 2003, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 CompuChem Quality Manual, Revision ~~12~~ 14 January 25, 2011, ~~August 3, 2009~~, plus revisions 06-14-2011

16.12 Sample Control SOP 4.1, "Receiving Samples"

16.13 Sample Control SOP 4.6, "Storing Samples"

16.14 DoD QSM Version 4.2, 10/25/2010 06-14-2011

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 –List of toxicity characteristic constituents and regulatory levels

17.2 Attachment 2 –Diagram of agitation apparatus

17.3 Attachment 3 –List of filter holders available

17.4 Attachment 4 –Worksheets for SPP -814 TCLP Waste Characterization Leachate

17.5 Attachment 5 – Balance Calibration Logbook

17.6 Attachment 6 – TCLP Laboratory Temperature Logbook

Attachment 1

Toxicity Characteristic Constituents and Regulatory Levels

TABLE II.2.—TOXICITY CHARACTERISTIC CONSTITUENTS AND REGULATORY LEVELS

EPA HW No.[1]	Constituent (mg/L)	CAS No.[2]	Chronic toxicity reference level (mg/L)	Regulatory level (mg/L)
D004	Arsenic	7440-38-2	0.05	5.0
D005	Barium	7440-39-3	1.0	100.0
D018	Benzene	71-43-2	0.005	0.5
D006	Cadmium	7440-43-9	0.01	1.0
D019	Carbon tetrachloride	56-23-5	0.005	0.5
D020	Chlordane	57-74-9	0.0003	0.03
D021	Chlorobenzene	108-90-7	1	100.0
D022	Chloroform	67-66-3	0.06	6.0
D007	Chromium	7440-47-3	0.05	5.0
D023	o-Cresol	95-48-7	2	[4] 200.0
D024	m-Cresol	108-39-4	2	[4] 200.0
D025	p-Cresol	106-44-5	2	[4] 200.0
D026	Cresol		2	[4] 200.0
D016	2,4-D	94-75-7	0.1	10.0
D027	1,4-Dichlorobenzene	106-46-7	0.075	7.5
D028	1,2-Dichloroethane	107-06-2	0.005	0.5
D029	1,1-Dichloroethylene	75-35-4	0.007	0.7
D030	2,4-Dinitrotoluene	121-14-2	0.0005	[3] 0.13
D012	Endrin	72-20-8	0.0002	0.02
D031	Heptachlor (and its hydroxide)	76-44-8	0.00008	0.008
D032	Hexachlorobenzene	118-74-1	0.0002	[3] 0.13
D033	Hexachloro-1,3-butadiene	87-68-3	0.005	0.5
D034	Hexachloroethane	67-72-1	0.03	3.0
D008	Lead	7439-92-1	0.05	5.0
D013	Lindane	58-89-9	0.004	0.4
D009	Mercury	7439-97-6	0.002	0.2
D014	Methoxychlor	72-43-5	0.1	10.0
D035	Methyl ethyl ketone	78-93-3	2	200.0
D036	Nitrobenzene	98-95-3	0.02	2.0
D037	Pentachlorophenol	87-86-5	1	100.0
D038	Pyridine	110-86-1	0.04	[3] 5.0
D010	Selenium	7782-49 2	0.01	1.0
D011	Silver	7440-22-4	0.05	5.0
D039	Tetrachloroethylene	127-18-4	0.007	0.7
D015	Toxaphene	8001-35-2	0.005	0.5
D040	Trichloroethylene	79-01-6	0.005	0.5
D041	2,4,5-Trichlorophenol	95-95-4	4	400.0
D042	2,4,6-Trichlorophenol	88-06-2	0.02	2.0
D017	2,4,5-TP (Silvex)	93-72-1	0.01	1.0
D043	Vinyl chloride	75-01-4	0.002	0.2

[1] Hazardous waste number.
[2] Chemical abstracts service number.
[3] Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level.
[4] If o-, m-, and p-cresol concentrations cannot be differentiated, the total cresol (D026) concentration is used. The regulatory level for total cresol is 200 mg/l.

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Attachment 2
Rotary Agitation

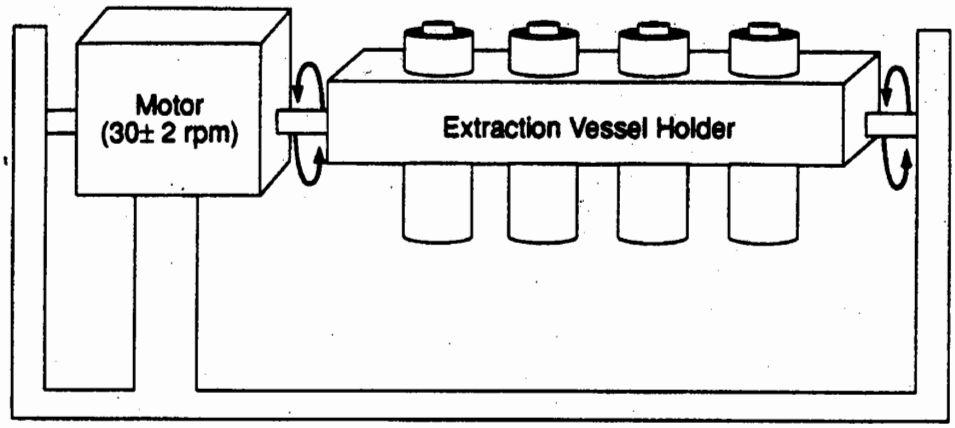

Figure 1. Rotary Agitation Apparatus

Attachment 3
Suitable Filter Holders1

Company	Location	Model	Size
Nuclepore Corporation	Pleasanton, California (800)882-7711	425910	142 mm
		410400	47 mm
Micro Filtration Systems	Dublin, California (415)828-6010	302400	142 mm
*Millipore Corporation	Bedford, Massachusetts (800-225-3384)	YT30142HW	142 mm
		XX1004700	47 mm

1 Any device capable of separating the liquid from the solid phase of the waste is suitable, provided that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic contaminants are of concern.

Suitable Filter Media

Company	Location	Model	Pore Size1
*Whatman Laboratory Products, Inc.	Clifton, New Jersey (201)773-5800	GFF	0.7

1 Nominal pore size

* Product used at CompuChem

Attachment 4

COMPUCHEM

ASSIGNED TO: _____ TCLP WASTE CHARACTERIZATION LEACHATE DATE EXTRACTED _____

Method 1311

EMPLOYEE ID: _____ -814

SAMPLE NUMBER	PRE-TEST pH VALUE		EXTRACTION FLUID AND VOL. (mL) ADDED		PARTICLE REDUCT. DONE (Y/N)	SAMPLE WEIGHT (g)	FINAL LEACH. pH VALUE	FINAL VOLUME (ml)	PERCENT SOLID	COMMENTS
	START	FINAL	1	2						
										TCLPBLK
										TCLPBLK

LOADED TUMBLER CALIB. CHECK (MUST BE 30 rpm ± 2 rpm)	
TUMBLER #:	CALC. RPM
(COUNT rpm FOR 30 sec. AND MULTIPLY NUMBER BY 2 TO CALCULATE RPM)	

ROTATION TIME ONLY

DATE/TIME STARTED _____/_____

DATE/TIME COMPLETED _____/_____

Room Temp. _____

Final Vol. Verified _____ Ext. Fluid 1 pH _____ (4.93 ± 0.05)

Reviewed By: _____

Ext. Fluid 2 pH _____ (2.88 ± 0.05)

[1] Enter volume (mL) of Extraction Fluid added into appropriate column, e.g., enter volume into column 1 if Extraction Fluid # 1 is used. Ensure that the fluid volume to sample weight ratio is 20:1

Manufacturer and lot # of reagent used _____ 3/15/00:mlj

Attachment 4 (continued)

COMPUCHEM a Division of Liberty Analytical
PERCENT SOLIDS
PRETEST

ASSIGNED TO _____ **DATE** ____

EMPLOYEE # _____ **-814**

CCN	WEIGHT OF FILTER (G)	WEIGHT OF BEAKER (g)	WEIGHT OF BEAKER SAMPLE (g)	WEIGHT OF EMPTY BEAKER RESIDUE	SAMPLE WEIGHT (g)	PRE-WEIGHT FILTRATE CONTAINER (g)	WEIGHT OF FILTRATE FILLED CONTAINER	WEIGHT OF FILTRATE	WEIGHT OF SOLIDS	% SOLID	WEIGHT OF DRIED FILTER	% DRY SOLIDS

	WEIGHT OF SAMPLE=T 100g SOLIDS (g)	ACTUAL SAMPLE WEIGHT (g)	WEIGHT OF FLUID NEEDED (g)	VOLUME EXT FLUID NEEDED (ml)	COMMENTS

6/20/01:dce

Attachment 5

CompuChem a division of Liberty Analytical Corp. Logbook 2 YY 1

Corning pH-30 Meter Calibration Log

Buffer 2.00	Buffer 4.00	Buffer 10.00	Date	Initials

If reading are not within ± 0.05 pH units of 2.00, 4.00, and 10.00, contact supervisor immediately.
The pH meter cannot be used if readings are not within ± 0.05 pH units of the buffer solution value.

Reviewed by: _____ **Date:** _____

SOP 3.2.1.4

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-1080 SOP Section #: 3.2.1.4 Revision #: 5

SOP Title: Digestion Block Preparation of Aqueous Samples for ICP Analysis of Total or Dissolved Metals by SW-846 and Standard Methods

Effective date: (QA fills in) 1/31/2011

- Procedure prepared by: Jan C Felch Date: 01/30/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Meier Date: 01-31-2011
- Reason for change: Annual Review; Removal of method 200.7
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, 12/96, 3010A; Standard Methods for the Examination of Water and Wastewater, 20th Edition, 1998

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -1080: Digestion Block Preparation of Aqueous Samples for ICP Analysis of Total or Dissolved Metals by SW-846 and Standard Methods

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Sample Preparation Procedure -1080: Digestion Block Preparation of Aqueous Samples for ICP Analysis of Total or Dissolved Metals by SW-846 and Standard Methods

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the digestion block preparation of aqueous samples for ICP determination of total or dissolved metals. The procedure is based on SW-846 Method 3010A **and** Standard Methods for the Examination of Water and Wastewater, 20th Edition Methods. This procedure incorporates a reduced sample aliquot of one half the normal aliquot (50 mL). All reagent volumes are proportionately reduced by one-half.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

To determine total metal concentrations, an accurately measured, well-mixed aliquot of the sample is treated with HNO_3 and HCl and heated in a continuously monitored digestion block to solubilize all metals. The resulting solution is cooled, filtered, and brought to volume for subsequent analysis by ICP.

Samples requiring dissolved metals analysis undergo an initial filtration and preservation, without digestion, prior to analysis.

Samples requiring pretreatment for acid-extractable metals are exposed to a more moderate digestion procedure.

3.0 Definitions

3.1 Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B). A minimum of seven sample replicates is required to calculate a

statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – µg/L for water

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 **DoD**-QSM – Department of Defense Quality Systems Manual

3.9 SC DHEC – South Carolina Department of Health and Environmental Control

3.10 NC DENR – North Carolina Department of Environment and Natural Resources

4.0 Interferences

4.1 Physical interferences are generally considered to be effects associated with the ICP analysis, especially with samples containing high dissolved solids and/or acid concentrations.

4.2 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects.

4.3 Interferences are highly dependent on matrix type and the specific analyte element. Further detail on managing interferences is found in Instrument Procedure SOP 309, "Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846."

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The Chemical Hygiene plan and MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Polypropylene digestion cups-50 mL

6.2 Watch glasses-ribbed

6.3 Environmental Express 36 and 54 well digestion block

6.4 membrane filter - 0.40 to 0.45 μm

7.0 Reagents and Standards

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20th Edition of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

7.2 Nitric acid (HNO_3) -concentrated, ultra-pure grade

7.2.1 Baker "Instra-analyzed" (BIA) or equivalent

7.2.2 Prepare a 1:1 solution by slowly adding the concentrated acid to water under a fume hood in the ratio of 100-mL acid to 100-mL reagent water.

7.2.2.1 Always add acid to water.

7.3 Hydrochloric acid (HCl) – concentrated, ultra-pure grade

7.3.1 Baker "Instra-analyzed" (BIA) or equivalent

7.3.2 Prepare a 1:1 solution by slowly adding the concentrated acid to water under the fume hood in the ratio of 100-mL acid to 100-mL reagent water.

7.4 Laboratory Control Sample (LCS) spiking solution

7.4.1 XCL-LCS-10X**R** - purchased, certified, aqueous standards for ICP laboratory control sample (LCS)

7.5 Matrix Spiking Solution

7.5.1 XCL-20 - purchased, certified, aqueous standard for spiking the MS/MSD for ICP.

7.6 TCLP STD-500 - purchased standard with concentrations at the regulatory limits

7.6.1 Used for spiking the TCLP LCS and TCLP MS/MSD

7.7 Silver-100 µg/mL, purchased, High Purity Standard

7.7.1 Used for spiking the TCLP LCS and TCLP MS/MSD

7.8 All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date **five** years from receipt. All lab prepared reagents must be assigned an expiration date one year from preparation.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Aqueous samples received for trace metals analyses are checked for acid preservation upon receipt. Metals samples not preserved at the time of collection with HNO_3 to a pH <2 may be preserved in-house. Samples must be preserved at least 24 hours prior to analysis.

8.3 Aqueous metals samples must be analyzed within 180 days from sampling in order to meet the method holding time.

8.4 Samples are obtained from the Custodian out of ambient storage. After preparation, they are returned to the Custodian for long-term storage and disposal.

8.4.1 TCLP extracts are stored in the cooler at 4 ^{o}C ± 2 ^{o}C.

9.0 Quality Control (QC)

9.1 Method Blank

9.1.1 Prepare a method blank for each batch of up to 20 samples.

9.2 Laboratory Control Sample

9.2.1 Prepare a LCS with each batch of up to 20 samples.

9.3 Matrix Spike and Matrix Spike Duplicate

9.3.1 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for each batch of up to 20 samples.

9.4 Duplicate

9.4.1 The NC DENR requires a 10% duplicate frequency which is met by the 5% MS/MSD frequency requirement.

9.5 All QC samples are digested at the same time as the batch samples.

10.0 Calibration and Standardization

Automated pipettes used to add standards to sample aliquots during sample preparation are calibration monthly. See SOP 9.1, "Calibrating Automatic Pipettes in the Inorganic Laboratory".

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". Complete the Metals Preparation Log (Attachment 1) as you proceed through the sample preparation. All traceability documentation must be recorded in the Standards/QC Preparation for Trace ICP log (Attachment 2)

11.1 Sample Preparation

11.1.1 Prepare the method blank using 50-mL reagent water.

11.1.2 Prepare the LCS by adding **5.0** mL of XCL-LCS-**10XR** 50 mL of reagent water.

11.1.2.1 Prepare the TCLP LCS by adding 0.5-mL of TCLP STD-500 and 0.50-mL of the 100-µg/mL Silver standard to 50-mL of reagent water.

11.1.3 Prepare the MS/MSD by adding 0.5-mL of XCL-20 solution to two separate 50-mL aliquots of the sample designated as the original.

11.1.3.1 Prepare the TCLP matrix spikes by adding 0.5-mL of TCLP STD-500 and 0.50-mL of the 100-µg/mL Silver standard to

two separate aliquots of the TCLP sample extract designated as the original.

11.2 Request samples scheduled to be prepped for the day from the Sample Custodian with a Sample Request Form (Internal Chain of Custody, COC) (Attachment 5). This form will accompany the raw samples when they are delivered to the preparation laboratory and remains with them until they are returned to the Sample Custodian. The preparation technician must sign and date the COC record.

11.3 Check the pH of each sample to ensure that each has been acidified to <2. If the sample has not been properly acidified, notify Customer Service who will contact the client for instructions on how to proceed.

11.4 Dissolved Metals

11.4.1 Samples not filtered and preserved at collection, are filtered and preserved to pH <2 with a 1:1 HNO_3 solution upon sample receipt. This process is recorded on the sample filtration worksheet (Attachment 3). **A waiting period of 24 hours is required after preservation.**

11.5 Digestion Procedure

11.5.1 Read the operating instructions for the block (Attachment 4) prior to turning it on for the first time. Turn on the digestion blocks. The set temperature is approximately 100 °-103 °C for the maintenance of 95 °C sample temperature.

11.5.2 Monitor the temperature of each block by placing a calibrated alcohol thermometer into on of the sample cups. Fill this cup with reagent water and place it in the block with the samples being digested. Periodically refill the cup with reagent water. Keep a thermometer in each block whenever samples are being digested. Record the temperature on the Metals Preparation Log.

11.5.3 Prepare labels for each sample. The label should contain the laboratory identifier and any QC designation.

11.5.4 For samples submitted to meet the regulatory requirements of the NC DENR, follow the steps in this section. This is Standard Methods for the Examination of Water and Wastewater, Method 3030C, "Preliminary Treatment for Acid-Extractable Metals". For all other samples, perform the Method 3010A digestion procedure starting with **S**ection 11.5.**5**.

11.5.4.1 Samples must be treated and filtered within 72 hour of sampling to minimize acid contact and digestion of any sediment in the sample. At collection in the field, samples are acidified by adding 5-mL concentrated HNO_3 to 1 liter sample.

11.5.4.2 Thoroughly mix the sample by shaking the container before removing an aliquot.

11.5.4.3 Transfer 50 mL of sample to a properly labeled digestion cup.

11.5.4.4 Add 2.5 mL of 1:1 HCl and cover with a ribbed watch cover.

11.5.4.5 Heat for 15 minutes in the digestion block.

11.5.4.6 Cool the samples and filter through a membrane filter (0.45 µm).

11.5.4.7 Dilute to the 50 mL mark with reagent water.

11.5.4.8 Relinquish samples to the ICP Chemist or ICP analysis storage area outside the ICP instrument lab.

11.5.4.9 Prepare the QC samples at the instrument bench following the directions provided in the Standards and QC Log and in **Section** 11.1.

11.5.5 Thoroughly mix the sample by shaking the container before removing an aliquot.

11.5.6 Transfer 50 mL of sample to a properly labeled polypropylene digestion cup. Add 1.5 mL concentrated nitric acid.

11.5.6.1 Prepare QC samples as described in Section 11.1. Add 1.5 mL of concentrated nitric acid.

11.5.7 Place the sample cups in the rack. Place ribbed watch covers on the cups and place into a 95° C digestion block. Monitor the digestion block temperature and record it on the Metals Preparation Log.

11.5.8 Heat samples for one hour with out boiling. Allow the samples to reduce but not to dry. At the end of the first hour, remove the cups from the digestion block. Remove and discard the watch cover.

11.5.9 Add 1.5 mL of concentrated nitric acid to each sample. Cover the samples with new non-ribbed watch covers and return the rack to the digestion block.

11.5.10 Gently reflux the samples, adding HNO_3 until the sample is clear **or** there is no change in appearance with continued refluxing. Remove the rack from the digestion block. Allow the samples to cool.

11.5.11 Add 5**.0** mL of 1:1 HCl and reflux the samples in the digestion block for another 15 minutes to dissolve any precipitate produced during the evaporation process.

Note: If samples have evaporated to less than about 30 mL, add 10 mL of reagent water prior to the addition of HCl. This will help to prevent the precipitation of elements, specifically Ag, that are sensitive to high concentrations of chlorides.

11.5.12 Adjust the sample volumes to 50 mL mark with reagent water. Attach screw-caps securely.

11.5.13 Deliver copies of the completed Sample Preparation Log with the samples to the ICP Chemist of ICP sample storage area. **Sign and date the internal digestion chain-of-custody (Attachment 6), relinquishing the samples.**

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has

established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal" requiring laboratory procedures for recycling waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, 3010A.

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080, 3030C, 3030F

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 NELAC Standards, **effective July,** 2003**, plus revisions**

16.6 QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.7 CompuChem Quality Manual, Revision **13**, **November 17, 2009**.

16.8 Sample Control SOP 4.6, "Storing Samples"

16.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 **– Sample** Metals Preparation Run Log

17.2 Attachment 2 **– Sample** Standards/QC Preparation Log

17.3 Attachment 3 **–** Sample Filtration Worksheet

17.4 Attachment 4 - Operating Instructions for Environmental Express Hot Blocks

17.5 Attachment 5 – **Example** Sample Request Form/Internal Chain-of-Custody

17.6 Attachment 6 – Example Internal Digestion Chain-of-Custody

Attachment 1

METALS/MERCURY PREPARATION LOG

Metals Method: 3010A / 6010B/C
Mercury Method: 7470A
Prepared By: _____

CompuChem
A Division Of
Liberty Analytical Corp.

Preparation Date: _____
Preparation Time: _____
Batch Number: _____

Blue Water

COMPUCHEM SAMPLE ID	CLIENT SAMPLE ID	SAMPLE TYPE	ICP Initial (mL)	ICP Final (mL)	HG Initial (mL)	HG Final (mL)	*Description Before	*Description After ICP	pH
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Mercury Standards and QC S0 S0.2 S0.5 S1 S5 S10 ICV ICB CRI CCV CCB

NOTE: All standards and QC for mercury were prepared at the same day as the associated samples. Refer to the attached Standard/QC Preparation Log, with date matching the above preparation date, for preparation method and instruction.

*** R=red, BL=blue, Y=yellow, G=green, O=orange, V=violet, W=white, CS=colorless, BR=brown, BK=black GY= grey, F=fine, M=medium, C=coarse, CL=clear, CY=cloudy, OP=opaque**

Record of Tempature/Time	
Hot Block Temp In:	C
Hot Block Temp Out:	C
Time Hg in water bath:	
Time Hg out water bath:	
Water Bath Temp In:	C
Water Bath Temp Out:	C

MS Reference
ICAP: 0.5 mL XCL-20
Ref #:
HG: 1.0mL 100ppb Hg
Ref#

LCS Reference
ICAP: 5.0 mL of XCL-10XR
Ref #:
HG: 3.0mL 100ppb Hg
Ref#

"The presence of the Chemist's/analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations requires documentation by the chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation."

Sample Verification Signature: _____
This signature corroborates that the sample idenification numbers appearing on the digestion cups are the same as those appearing on the field sample containers.

Reviewed By: _____
Date: _____

Shading has been applied to unused portions of this worksheet as a substitute for 'Z-out.

JCF 120209

Note: This page subject to change without notice.

Attachment 2

Standards/QC Preparation Log for ICP Metals/Mercury Page 1 of 3 **Case/SDG:**_____
COMPUCHEM a division of Liberty Analytical Corp. Worksheet 1Q (1) 2

REAGENTS	REF#[1]	MANUFACTURER	LOT#	EXPIRATION DATE
SULFURIC ACID (H_2SO_4)	0H13010	CALEDON	74387	08/15
NITRIC ACID (HNO_3)	0J27007	JT BAKER	J29049	10/15
POTASSIUM PERMANGANATE ($KMnO_4$)	0F30018	JT BAKER	J04613	06/15
HYDROCHLORIC ACID (HCL)	0L21024	CALEDON	74606	12/15
POTASSIUM PERSULFATE ($K_2S_2O_8$)	0F30018	JT BAKER	H44465	06/15
HYDROGEN PEROXIDE (H_2O_2)	0I02006	CALEDON	72923	05/11
STANDARDS				
XCL 20R	0I15003	HIGH PURITY	1025219	09/13/11
XCL LCS 10XR	0H03016	HIGH PURITY	1021106	08/02/11
ERA PPT (540)	0G07001	ERA	540	07/07/15
EPA LCS (0405)	0E20016	EPA	0405	05/17/15
ICV-1 (0307)	0E20013	EPA	0307	05/17/15
ILM05.3 MATRIX SPIKE	0B23010	HIGH PURITY	1004722	02/18/11
SILVER (Ag) 100ppm	0I16001	HIGH PURITY	1013221	12/14/11
TCLP 500	0L16002	HIGH PURITY	1034422	12/14/11
ARSENIC (As) 1000ppm	0L01020	HIGH PURITY	1021405	06/01/12
MERCURY (Hg) 20ppm	0F16001	HIGH PURITY	1015809	06/07/11
ICV-5 (0508) Hg	0E20020	EPA	0508	05/17/15

[1]Ref#=Logbook ID, Page number, and item number from Materials Receipt Log
[2]Standard Lot Number consist of standard ID, date prepared, and initials of preparer

Pb 1000ppm	0L01019	HIGH PURITY	1026448	06/01/12

STANDARD SOLUTIONS PREPARED BY:_____ DATE:_____

ENTERED BY:_____ DATE:_____

REVIEWED BY:_____ DATE:_____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this worksheet attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

12/03/07:RM

Note: This page subject to change without notice.

Attachment 2 (continued)

Standards/QC Preparation Log for ICP Metals/Mercury Page 2 of 3 **Case/SDG:**_____
COMPUCHEM a division of Liberty Analytical Corp. Worksheet 1Q (1) 2

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS
Hg 100ppb-01-07-11-DD	Place in a 100mL volumetric flask: 0.5mL of 20ppm Hg stock solution 2.5mL of concentrated HNO_3 Baker instra-analyzed Trace Metals grade or equivalent Dilute to 100mL volume with reagent water (**PREPARE DAILY**)
$K_2S_2O_8$- 12-13-10-DD Potassium Persulfate	Place in a 1000mL Erlenmeyer to a re-pipettor bottle (**PREPARE AS NEEDED**)
$KmnO_4$. 12-20-10 –DD Potassium Permanganate	Place in a 2000mL volumetric flask: 100.0g of $KmnO_4$ Baker instra-analyzed Trace Metals grade or equivalent Dilute to 2000mL with reagent water (**PREARE AS NEEDED**)
HNO_3- 12-16-10 - NB Nitric Acid	Place in a 2000mL volumetric flask: 500mL of Deionized water 1000mL of concentrated HNO_3 Baker instra-analyzed Trace Metals grade or equivalent Dilute to 2000mL with reagent water (**PREPARE AS NEEDED**)
HCl- 12-16-10 - NB Hydrochloric Acid	Place in a 2000mL volumetric flask: 500mL of Deionized water 1000mL of concentrated HCl Baker instra-analyzed Trace Metals grade or equivalent Dilute to 2000mL with reagent water (**PREPARE AS NEEDED**)
Aqua Regia- 01-07-11 - DD	Place in a 1000mL container: 600mL of Concentrated HCL 200mL of Concentrated HNO3 **(PREPARE AS NEEDED)**

[1]Ref#=Logbook ID, Page number, and item number from Materials Receipt Log
[2]Standard Lot Number consist of standard ID, date prepared, and initials of preparer

STANDARD SOLUTIONS PREPARED BY:_____ DATE:_____

ENTERED BY:_____ DATE:_____

REVIEWED BY:_____ DATE:_____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this worksheet attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

12/03/07:RM

Note: This page subject to change without notice.

Attachment 2 (continued)

Standards/QC Preparation Log for ICP Metals/Mercury Page 3 of 3 **Case/SDG:**_____
COMPUCHEM a division of Liberty Analytical Corp. Worksheet 1Q (1) 2

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS	Resultant Analyte Concentration
ICV-5- 01-07-11-DD	Pipet into a 300mL BOD Bottle 1.0mL of ICV-5 and 99mL of reagent water. Digest according to sample preparation instructions	4.0µg/L
S0/ICB/CCB- 01-07-11-DD	Pipet into a 300mL BOD Bottle 100mL of reagent water Digest according to sample preparation instructions	0.0µg/L
S0.2/CRA/CRI- 01-07-11-DD	Pipet into a 300mL BOD Bottle 0.2mL of Hg 100ppb and 99.8mL of reagent water Digest according to sample preparation instructions	0.2µg/L
S0.5- 01-07-11-DD	Pipet into a 300mL BOD Bottle 0.5mL of Hg 100ppb and 99.5mL of reagent water Digest according to sample preparation instructions	0.5µg/L
S1.0- 01-07-11-DD	Pipet into a 300mL BOD Bottle 1.0mL of Hg 100ppb and 99mL of reagent water Digest according to sample preparation instructions	1.0µg/L
S5.0/CCV 01-07-11-DD	Pipet into a 300mL BOD Bottle 5.0mL of Hg 100ppb and 95mL of reagent water Digest according to sample preparation instructions	5.0µg/L
S10.0- 01-07-11-DD	Pipet into a 300mL BOD Bottle 10mL of Hg 100ppb and 90mL of reagent water Digest according to sample preparation instructions	10.0µg/L

[1]Ref#=Logbook ID, Page number, and item number from Materials Receipt Log
[2]Standard Lot Number consist of standard ID, date prepared, and initials of preparer

STANDARD SOLUTIONS PREPARED BY:_____ DATE:_____

ENTERED BY:_____ DATE:_____

REVIEWED BY:_____ DATE:_____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this worksheet attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

12/03/07:RM

Note: This page subject to change without notice.

Attachment 3

Metals Filtration Preparation Log
Metals Method : Filtration
CompuChem a Division of Liberty Analytical Corp.

Case/SDG:_____ Prepared by:_____ Date:_____

#	CCN (Lab ID)	Client ID	Date Rec'd	FILTRATION PERFORMED (CHECK)	pH
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

Reagent Manufacturer & Lot #'s:

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If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 4

Operating Instructions

The HOT BLOCK

Catalog Number:

36 well Digestion Block	SC100
Polypropylene Digestion Cups,500/pk.	SC500
18 Cup Tray	SC200

Specifications

Size: 15" X 15"
Weight: 43 lb.
Voltage: 120 VAC
Current: 5 amps maximum
Well Size: 3.14" X 2.75"
Cup Capacity: 50mL
Max. Cup Temperature: 130°C
Temperature Range: Ambient - 165°C
Controller: Ogden ETR -3020
Temperature Stability: ± 0.1°C
Temperature Uniformity: ± 1°C
Time to Heatup (empty): 13 minutes
Time to Heatup (loaded): 25 minutes

Environmental Express, Ltd..
490 Wando Park Blvd.
Mt. Pleasant, SC 29464
800-343-5319 (o)
803-881-3964 (f)
www. envexp.com

The Environmental Express® Hot Block™ provides an elegant and efficient method of digesting and storing water, wastewater, soil and sludge samples for metals analysis. This innovative system allows samples to be digested in a corrosion free environment. In addition, samples are handled in a small area with minimal radiant heat loss.

Construction
The Hot Block contains almost no metal components. Only the thermocouple (buried and sealed in the graphite), and the controller contacts (sealed with acrylic), are metal. A nitride coating prevents oxidative agents from attacking the integrity of graphite surfaces, and standard digestion agents such as HCL and HNO3 demonstrate no adverse effect on graphite or the fire resistant, box construction material, Kydex®. Box fasteners are PVC. The internal assembly is encased with 1" of thermal insulation.

Location
Locate the Hot Block under a fume hood with a minimum face velocity of 100 fpm, and allow a minimum of 2" of space on all sides.

Temperature Setting
The preset factory "Control Point" temperature is 115°C. Tests have shown that acid solutions in the polypropylene digestion cups with a Control Point temperature of 115°C will achieve 95°C at 760mm of Mercury (sea level). If digestions are being performed at higher elevations it may be necessary to reduce the set point temperature.
Note: The maximum use temperature of the polypropylene cup is 130° C.

To Adjust Temperature
(1) Turn the Hot Block on and wait until the display shows the current block temperature.

(2) Press the left arrow key. The display will show the Control Point temperature. The flashing digit can be changed with the up or down arrow keys.
(3) To change to another flashing digit, press the left arrow key.
(4) When the proper Control Point is reached, press the set key to return to the current temperature display.

Temperature Limit
The controller of the Hot Block has an upper control limit of 150°C. This upper control limit may be changed if desired (see the accompanying controller manual). There is a non-serviceable safety fuse set at 168°C. If the block temperature rises above 168°C, this fuse will melt. (A complete guide to the controller is included with your Hot Block). It is important to note that different liquids may achieve different temperature at the same Hot Block temperature setting. This is due to different vapor pressures of different liquids. When performing standard acid digestions of environmental samples, samples with high oil content will achieve higher temperatures than aqueous samples.

Maintenance
The Hot Block has no serviceable parts. Any service inquiries should be directed to Environmental Express. The unit should be cleaned regularly with water to remove acid residue. Acid that is spilled directly in the digestion wells should be neutralized and removed.

Warranty
This product is warranted against defects in materials and workmanship when used in accordance with the applicable instructions for a period of one year from the date of purchase. The warranty remedy is limited to product repair. If Environmental Express is unable to repair the Hot Block, the customer may, at his/her option receive a replacement unit or a full refund. **Operating the Hot Block at temperatures higher than 160°C will void the warranty.**

Attachment 5

Internal COC Metals/Mercury Samples

5/5/2010

Metals Internal Chain of Custody Sheet

Batch: 0060346 **Status:** Batched **Analysis:** 6010B METALS

Lab Id	Client_Id	Received	Container	Extraction	Preservative	Matrix	Due Date	Cust Date
1005180-01 B	SS41-0-1	05/21/10	4a_2OZ WM Glass, cool	EPA 3050B	Cool 4°C	Soil	6/10/2010	5/28/2010
1005180-02 B	SB41-1-2	05/21/10	4a_2OZ WM Glass, cool	EPA 3050B	Cool 4°C	Soil	6/10/2010	5/28/2010
1005180-03 B	SS35-0-1	05/21/10	4a_2OZ WM Glass, cool	EPA 3050B	Cool 4°C	Soil	6/10/2010	5/28/2010
1005180-04 B	SB35-2-3	05/21/10	4a_2OZ WM Glass, cool	EPA 3050B	Cool 4°C	Soil	6/10/2010	5/28/2010

Relinquished By	Date/Time	Received By	Date/Time
Cooler 1	6.4.10 0833	Diane Byrd	6.4.10 0833
Diane Byrd	6.4.10 0833		

1

Note: This page subject to change without notice.

Attachment 6

Internal Digestion Chain-of-Custody

INTERNAL DIGESTION COC

Printed: 5/28/2010 2:13:28PM

Lab Number	Client ID	Sample Type	

Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date

Page 1 of 1

Note: This page subject to change without notice.

SOP 3.2.1.5

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-240 SOP Section #: 3.2.1.5 Revision #: 8

SOP Title: Digestion Block Preparation of Soled Samples for ICP Determination of Total Metals by SW-846 Method 3050B

Effective date: (QA fills in) 01-28-2011

- Procedure prepared by: Jon C Feldh Date: 01/12/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob McEver Date: 01-25-2011
- Reason for change: Annual Review; Addition of DoD procedures and protocols
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III 12/96, Method 3050B; Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -240: Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 Method 3050B

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Sample Preparation Procedure -240: Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 Method 3050B

1.0 Scope and Application

This method describes the digestion procedure for the preparation of solid samples, including soil, sediment, sludge, and biota, for Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). Sample volume and reagent volumes do not differ from the volumes used in the traditional beaker preparation procedure. This procedure is based upon SW-846 Method 3050B.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A representative 0.95-1.9 gram sample aliquot is digested with repeated additions of nitric acid and hydrogen peroxide. Biota samples must first be homogenized. Hydrochloric acid is added to the initial digestate and the sample is refluxed. The digestate is diluted to a final volume of 100 **mL**.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false

negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – mg/Kg

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM does not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 D**o**D-QSM – Department of Defense Quality Systems Manual

3.9 Extraction Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

4.0 Interferences

4.1 Chemical interferences are characterized by molecular compound formation, ionization effects, and solute vaporization effects.

4.2 Interferences are highly dependent on matrix type and the specific analyte element. Further detail on managing interferences is found in Instrument Procedure SOP 309, "Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 **Methods 6010B and 6010C**."

5.0 Safety

5.1 Care should be taken when handling acids. Always use acids under approved fume hoods. Always pour acid into water.

5.2 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 In this prep method, the 50 mL cup requires the technician to closely monitor sample reaction to reagents in order to avoid sample overflow.

5.4 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and MSDS**s** are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 Polypropylene digestion cups – 50 mL

6.2 Ribbed polypropylene watch glasses

6.3 Environmental Express 36 and 54 well digestion blocks

6.4 Whatman No. 41 filter paper or equivalent.

6.5 Analytical balance--capable of accurately weighing to the nearest 0.01 g.

6.6 Volumetric flasks – 100 mL

6.7 Blender for homogenizing biota samples.

6.8 Small plastic funnel

7.0 Reagents and Standards

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date **five** years from receipt. All lab prepared regents must be assigned an expiration date one year from preparation.

7.2 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (18th and 19th Editions of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

7.3 Nitric acid (HNO_3) – concentrated, ultra-pure reagent.

7.3.1 All concentrated nitric acid used in preparations should be Baker "Instra-analyzed" (BIA) or an equivalent.

7.3.2 1:1 HNO_3

7.3.2.1 Prepare a 1:1 solution by slowly adding one part acid to one part reagent water.

7.4 Hydrochloric acid (HCl) – concentrated, ultra-pure reagent

7.4.1 All concentrated hydrochloric acid used in preparations should be Baker "Instra-analyzed" (BIA) or an equivalent.

7.4 Hydrogen peroxide (30%) - purified and preserved with tin.

7.5 ERA PPT (540) Soil, purchased, certified LCS soil standard.

7.5.1 This standard will be used as the Laboratory Control Sample (LCS).

7.6 High-Purity Standards XCL-20, purchased, certified aqueous standard

7.6.1 For use in spiking the duplicate matrix spikes

7.7 Dry ice for the homogenization of biota

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, “Receiving Samples” and 4.6, “Storing Samples.” Sample holding times are also listed.

8.3 Soil samples are stored under refrigeration at 4 ºC ± 2 ºC.

8.4 All samples must be digested and analyzed within 180 days of collection.

8.5 Samples are obtained from the Custodian out of cold storage. They should be allowed to come to room temperature prior to sample preparation. After preparation, they are returned to the Custodian and placed in the cooler at 4 ºC ± 2 ºC for long-term storage and disposal.

8.6 After analysis, the residual digestates are boxed up and stored in the ICP laboratory and Sample Control. After three months they are disposed of in the acid waste stream.

9.0 Quality Control (QC)

9.1 Method Blank

9.1.1 Prepare a blank for each digestion batch of up to 20 samples.

9.2 Laboratory Control Sample

9.2.1 Prepare a LCS for each preparation batch of up to 20 samples.

9.3 Matrix Spike/Matrix Spike Duplicate

9.3.1 Prepare a matrix spike (MS) and matrix spike duplicate (MSD) for each sample batch of up to 20 samples.

9.4 Duplicate

9.4.1 For NC DENR, duplicates are required at a 10% frequency. The duplicate frequency is satisfied with the duplicate matrix spikes.

10.0 Calibration and Standardization

10.1 Ensure that the balance has been calibrated for the day before weighing out samples. Refer to Quality Control SOP 13.16, "Top Loading Balance Calibration and Maintenance."

10.2 Automated pipettes used to add standards to sample aliquots during sample preparation are calibration monthly. See SOP 9.1, "**Verifying** Automatic Pipettes **Calibrations**".

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6**:** "Proper Documentation Procedures".

11.1 Refer to the operating instructions for the block (Attachment **1**) prior to turning it on for the first time. Turn on the digestion blocks. The set temperature for our elevation is approximately 100°-103 °C for the maintenance of 95 °C sample temperature.

11.2 Monitor the temperature of each block during sample digestion using a calibrated alcohol thermometer. Place a digestion cup filled with reagent containing a thermometer in each block digestion. Periodically refill the cups with reagent water. Record the temperature on the Metals/Mercury Preparation Log (Attachment 2).

11.3 Request samples to be prepared for the day from the Sample Custodian using properly completed Sample Request Form **[Internal Chain-of-Custody – (COC)** Attachment 3**].** The preparation technician must sign and date the COC record. The form accompanies the samples until preparation is completed and they are returned to the Custodian.

11.4 Prepare labels for each sample. The label should contain the laboratory-assigned sample ID and any QC designation.

11.5 Soil Sample Preparation

11.5.1 Prepare the method blank using a **clean** solid matrix when available or an empty digestion cup to which the sample volumes of all reagents are added as the other samples in the batch.

11.5.2 Prepare the LCS adding 1.0 g of ERA PPT (540) Soil into a digestion cup. For MPCA samples, prepare two duplicate LCSs.

11.5.3 Prepare the MS/MSD by weighing two additional 0.95-1.9 g aliquots of the sample designated as the original into separate digestion cups. Add 1.0 mL of XCL-20 to each sample aliquot.

11.5.4 Allow field sample to equilibrate to room temperature. Discard any foreign objects such as sticks, stones/rocks, and leaves from the sample container. Mix the sample, using a clean spatula, inside the sample container breaking any clumps. If necessary, sieve the sample using USS #10 sieve.

11.5.5 Prepare the field sample by weighing a 0.95 - 1.9 gram representative sample aliquot into a 50 mL digestion cup. Record the weight of the aliquot on the preparation log (Attachment **2**).

11.6 Biota Sample Preparation

11.6.1 If the same biota samples are being prepared for other analyses that require homogenizing, an aliquot of the sample must be taken after homogenizing (but before drying).

11.6.2 Weigh and record the entire sample weight. Freeze the sample at –10 ^{o}C for 2 hours prior to homogenization.

11.6.3 Pack the blender below the blades with dry ice. Cut the frozen sample into small pieces and place them in the blender. Cover with dry ice and allow them to sit for one minute to refreeze.

11.6.4 Blend the sample. Pour the blended sample into a sample container and place the container in the freezer at –10 ^{o}C overnight. Leave the lid loose so the dry ice can sublimate overnight. The sample will then be ready for the preparation procedure.

Note: Do not air dry the sample.

Note: Biota sample instructions do not pertain to Ohio VAP work.

11.7 Digestion Procedure

11.7.1 Add 10 **mL** of 1:1 nitric acid to the sample in the digestion cup. Swirl to mix.

11.7.2 Place the digestion cups in the sample rack and place the rack into the digestion block. Place a watch glass on the digestion cup.

11.7.3 Heat at 95 **°C** for 10-15 minutes without boiling. Remove the sample rack from the digestion. Allow the sample to cool.

11.7.4 Add 5 **mL** concentrated nitric acid, and replace the watch glass. Reflux the sample for another 30 minutes. Remove the sample cup rack from the block. Allow the samples to cool.

11.7.4.1 If oxidation of the sample by the HNO_3 occurs (indicated by the generation brown fumes), add HNO_3 in 5 **mL** volumes until no brown fumes are generated by the sample.

11.7.5 Place the sample rack in the digestion block and heat the samples for 30 minutes without boiling. <u>Do not allow the sample to dry.</u>

11.7.6 Remove the sample rack for the digestion block and allow the sample to cool.

11.7.7 After the samples have cooled, add 2 **mL** of reagent water and 3 **mL** of 30% hydrogen peroxide to the sample. Place the sample rack back in the digestion block to warm the samples and start the peroxide reaction. Watch the samples for vigorous reaction to the peroxide. Care should be taken to avoid losses due to vigorous effervescence. Continue adding

30% peroxide in 1 **mL** volumes with heating until effervescence subsides, remove from the digestion block, and cool the solution.

Note: Do not add more than a total of 10 **mL** of 30% peroxide to each sample.

11.7.8 Add 10mL of concentrated HCL to the sample and replace the watch glass. Place the sample rack back in the digestion block and reflux for 15 minutes.

11.7.9 Remove the sample rack and allow the samples to cool.

11.8 Sample Filtration

11.8.1 Place a Whatman #41 paper in a clean funnel that rests atop a properly cleaned 100 **mL** volumetric flask (Class A). Prepare the filter and glassware with 1:1 nitric acid followed by a reagent water rinse.

11.12.1.1 See Glassware Preparation SOP 10.2, "Preparing Glassware for the Inorganics Laboratory" for glassware cleaning procedures.

11.8.2 Pour the contents of the sample cup into the filter. Thoroughly rinse the insides of the sample cup into the filter using reagent water. Ensure that the rinse volume does not cause the sample to rise above the top of the filter.

11.9 Dilute the digestate in the flask to a final volume of 100 **mL** with reagent water.

11.10 Place the sample label from the digestion cup on a 100 **mL** sample cup and transfer the sample to the cup. The sample **digestate** is now ready for analysis.

11.11 Deliver the samples and copies of the preparation worksheet to the ICP analyst or ICP lab sample storage area. Sign and date the internal **digestion** chain-of-custody **(Attachment 4)** relinquishing the samples.

11.12 The following <u>optional</u> procedure may be used to improve solubility and recoveries for antimony, barium, lead, and silver.

11.12.1 Add 2.5 **mL** concentrated HNO_3 and 10 **mL** concentrated HCl to a 0.95-1.9 gram sample (wet weight) and cover with a watch glass. Place the sample in the heating block and heat for 15 minutes at 95 ^{o}C.

11.12.2 Filter the digestate through Whatman No. 41 filter paper and collect the filtrate in a 100 **mL** volumetric flask. Wash the filter paper, while still in the funnel with no more than 5 **mL** of hot (~95 ^{o}C) HCl, then with 20 **mL** of hot (~95 ^{o}C) reagent water. Collect the washings in the same 100 **mL** volumetric flask.

11.12.3 Remove the filter and residue from the funnel, and place them back in the vessel. Add 5 **mL** of concentrated HCl, place the vessel back on the heating block, and heat at 95 ^{o}C ± 5 ^{o}C until the filter paper dissolves. Remove the vessel from the heating block and wash the cover and sides with reagent water. Filter the residue and collect the filtrate in the same 100 **mL** volumetric flask. Allow the filtrate to cool, then dilute to volume.

Note: High concentrations of metal salts with temperature sensitive solubility can result in the formation of precipitates upon cooling of primary and/or secondary filtrates. If precipitation occurs in the flask upon cooling, do not dilute to volume.

11.12.4 If a precipitate forms on the bottom of the flask, add up to 10 **mL** of concentrated HCl to dissolve the precipitate. After the precipitate is dissolved, dilute to volume with reagent water.

11.12.5 The sample is now ready for analysis. Follow steps in **Sections** 11.10 and 11.11

12.0 Data Analysis and Calculations

12.1 Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.2 Sample results are based upon dry weight of the sample. The dry weight of the sample must be determined according to Sample Preparation Procedure –143, **"Undecanted Percent** Moisture Determination **in Soil/Sediment/Sludge by CLP** and **SW-846".** This value used as a correction factor for the determination of the dry weight value.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal" requiring laboratory procedures for recycling waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 3050B.

16.2 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.3 Quality Control SOP 13.4, "Numerical Data Reduction"

16.4 NELAC Standards, **effective July,** 2003**, plus revisions**

16.5 QA/G6: Guidance for the Preparing Standard Operating Procedures (SOPs) EPA/600/ B-07/001, April 2007.

16.6 CompuChem Quality Manual, Revision **13**, **November 17**, **2009**

16.7 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 – Digestion Block Operating Instructions

17.2 Attachment 2 – **Example** Metals/Mercury Preparation Log

17.3 Attachment 3 – **Example** Internal **Digestion Chain-of-**Custody

17.4 Attachment 4 – Example Internal Digestion Chain-of-Custody

Attachment 1

Operating Instructions

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Cup Capacity: 50mL
Max. Cup Temperature: 130°C
Temperature Range: Ambient - 165°C
Controller: Ogden ETR -3020
Temperature Stability: ± 0.1°C
Temperature Uniformity: ± 1°C
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The Environmental Express® Hot Block™ provides an elegant and efficient method of digesting and storing water, wastewater, soil and sludge samples for metals analysis. This innovative system allows samples to be digested in a corrosion free environment. In addition, samples are handled in a small area with minimal radiant heat loss.

Construction
The Hot Block contains almost no metal components. Only the thermocouple (buried and sealed in the graphite), and the controller contacts (sealed with acrylic), are metal. A nitride coating prevents oxidative agents from attacking the integrity of graphite surfaces, and standard digestion agents such as HCL and HNO3 demonstrate no adverse effect on graphite or the fire resistant, box construction material, Kydex®. Box fasteners are PVC. The internal assembly is encased with 1" of thermal insulation.

Location
Locate the Hot Block under a fume hood with a minimum face velocity of 100 fpm, and allow a minimum of 2" of space on all sides.

Temperature Setting
The preset factory "Control Point" temperature is 115°C. Tests have shown that acid solutions in the polypropylene digestion cups with a Control Point temperature of 115°C will achieve 95°C at 760mm of Mercury (sea level). If digestions are being performed at higher elevations it may be necessary to reduce the set point temperature.
Note: The maximum use temperature of the polypropylene cup is 130° C.

To Adjust Temperature
(1) Turn the Hot Block on and wait until the display shows the current block temperature.

(2) Press the left arrow key. The display will show the Control Point temperature. The flashing digit can be changed with the up or down arrow keys.
(3) To change to another flashing digit, press the left arrow key.
(4) When the proper Control Point is reached, press the set key to return to the current temperature display.

Temperature Limit
The controller of the Hot Block has an upper control limit of 150°C. This upper control limit may be changed if desired (see the accompanying controller manual). There is a non-serviceable safety fuse set at 168°C. If the block temperature rises above 168°C, this fuse will melt. (A complete guide to the controller is included with your Hot Block). It is important to note that different liquids may achieve different temperature at the same Hot Block temperature setting. This is due to different vapor pressures of different liquids. When performing standard acid digestions of environmental samples, samples with high oil content will achieve higher temperatures than aqueous samples.

Maintenance
The Hot Block has no serviceable parts. Any service inquiries should be directed to Environmental Express. The unit should be cleaned regularly with water to remove acid residue. Acid that is spilled directly in the digestion wells should be neutralized and removed.

Warranty
This product is warranted against defects in materials and workmanship when used in accordance with the applicable instructions for a period of one year from the date of purchase. The warranty remedy is limited to product repair. If Environmental Express is unable to repair the Hot Block, the customer may, at his/her option receive a replacement unit or a full refund. **Operating the Hot Block at temperatures higher than 160°C will void the warranty.**

Attachment 2

METALS/MERCURY PREPARATION LOG

Metals Method: 3050B / 6010B
Mercury Method: 7471B
Prepared By: _____

CompuChem
A Division Of Liberty Analytical Corp.

Preparation Date: _____
Preparation Time: _____
Batch Number: _____

Blue Soil

COMPUCHEM SAMPLE ID	CLIENT SAMPLE ID	SAMPLE TYPE	ICP Initial (g)	ICP Final (mL)	HG Initial (g)	HG Final (mL)	*Description Before	*Description After ICP	
							/	/	
							/	/	
							/	/	
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Mercury Standards and QC S0 S0.2 S0.5S1 S5 S10 ICV ICB CRI CCV CCB

NOTE: All standards and QC for mercury were prepared at the same day as the associated samples. Refer to the attached Standard/QC Preparation Log, with date matching the above preparation date, for preparation method and instruction.

Record of Tempature/Time	
Hot Block Temp In:	C
Hot Block Temp Out:	C
Time Hg in water bath:	
Time Hg out water bath:	
Water Bath Temp In:	C
Water Bath Temp Out:	C

MS Reference
ICAP: **1.0mL XCL-20**
Ref #
HG: **1.0mL 100ppb Hg**
Ref#

LCS Reference
ICAP: **1.0g ERA PPT (540)**
Ref #
HG: **0.2g ERA PPT (540)**
Ref#

*** R=red, BL=blue, Y=yellow, G=green, O=orange, V=violet, W=white, CS=colorless, BR=brown, BK=black GY= grey, F=fine, M=medium, C=coarse, CL=clear, CY=cloudy, OP=opaque**

"The presence of the Chemist's/analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations requires documentation by the chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation."

Sample Verification Signature: _____
This signature corroborates that the sample idenification numbers appearing on the digestion cups are the same as those appearing on the field sample containers.

Reviewed By: _____
Date: _____

Shading has been applied to unused portions of this worksheet as a substitute for 'Z-out.

Balance ID#: _____

JCF 102610

Note: This page subject to change without notice.

Attachment 3

1/25/2011

Metals Internal Chain of Custody Sheet

Batch: **Status:** **Analysis:**

Lab Id	Client_Id	Received	Container	Extraction	Preservative	Matrix	Due Date	Cust Date

Relinquished By	Date/Time	Received By	Date/Time
Relinquished By	Date/Time	Received By	Date/Time
Relinquished By	Date/Time	Received By	Date/Time
Relinquished By	Date/Time	Received By	Date/Time

1

Note: This page subject to change without notice.

Attachment 4

INTERNAL DIGESTION COC

Printed: 5/28/2010 2:13:28PM

Lab Number	Client ID	Sample Type	

Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date

Page 1 of 1

Note: This page subject to change without notice.

SOP 3.2.1.6

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 309 SOP Section #: 3.2.1.6 Revision #: 19

SOP Title: Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 Methods 6010B and 6010C

Effective date: (QA fills in) 10/18/11

- Procedure prepared by: Judith Nozzi Date: 10/17/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date:
  Jad 10/17/11 [illegible] 101811
- Reason for change: Annual review and Addition of LIMS Information
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Ed, Update III, 12/96, Method 6010B and Update IV, 2/2007, Method 6010C, Rev. 3; DoD-QSM, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 309: Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 Methods 6010B and 6010C

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If words above are not highlighted, this is an uncontrolled copy of this document.

Instrument Procedure 309: Inductively Coupled Plasma Atomic Emission Spectroscopy by SW-846 Methods 6010B and 6010C

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the determination of elements in solution including metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP/AES). This SOP is applicable to a large number of metals and wastes. All matrices, including groundwater, aqueous samples, EP extracts, industrial wastes, soils, sludges, sediments, TCLP leachates, and other solid wastes, require digestion prior to analysis.

Methods 6010B and 6010C are applicable to the elements listed in Attachment 5. The elements determined for TCLP analysis are listed in Section 3.6. Detection limits, sensitivity, and optimum ranges of the metals will vary with the matrices and model of spectrometer. The data shown in these attachments are provided from clean aqueous samples.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies. Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences.

2.0 Summary of Method

Prior to analysis, samples must be solubilized or digested following Sample Preparation Procedure –1080, "Digestion Block Preparation of Aqueous Samples for the ICP Determination of Total or Dissolved Metals by SW-846, MCAWW, and Standard Methods" or Sample Preparation Procedure –240, "Digestion Block Preparation of Solid Samples for ICP Determination of Total Metals by SW-846 Method 3050**B**" TCLP samples must be leached following Sample Preparation Procedure –814, "Toxicity Characteristic Leaching Procedure (TCLP) **by SW-846 Method 1311**" after which the leachates are digested as liquid samples using the TCLP spiking levels.

Methods 6010B and 6010C are executed by the laboratory using the simultaneous, multielemental determination of elements by ICP. The method measures element-

emitted light by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the lines are monitored by photomultiplier tubes. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate corrections made. Three exposures are averaged to obtain a final result.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL $<$ LOD $\leq$ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported between the DL and the LOQ must be flagged as estimated values.

3.6 Instrument Detection Limits (IDLs) – IDLs are helpful to evaluate the instrument noise level and response changes over time for each analyte. They aren't to be confused with the lower limit of quantitation and should not be used in establishing that limit.

3.8 Reporting Units – µg/L for water and mg/Kg for soil

3.9 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.10 Toxicity characteristics constituents and regulatory levels for TCLP

Constituent (mg/L)	**Chronic toxicity reference level (mg/L)**	**Regulatory level (mg/L)**
Arsenic	0.05	5.0
Barium	1.0	100.0
Cadmium	0.01	1.0
Chromium	0.05	5.0
Lead	0.05	5.0

Constituent (mg/L)	Chronic toxicity reference level (mg/L)	Regulatory level (mg/L)
Mercury	0.002	0.2
Selenium	0.01	1.0
Silver	0.05	5.0

3.11 SC DHEC – South Carolina Department of Health and Environmental Control

3.11.1 Note: For samples submitted to meet the regulatory requirements of the SC DHEC, analyses have to be performed using Method 6010C.

3.12 **DoD**-QSM – Department of Defense Quality Systems Manual

3.13 **Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.**

3.14 **LIMS – Laboratory Information Management System**

4.0 Interferences

4.1 Spectral Interferences are caused by: (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) stray light from the line emission of high-concentration elements. Spectral overlap can be compensated for by computerized correction of the raw data after monitoring and measuring the interfering element. Unresolved overlap requires selection of an alternate wavelength. Background contribution and stray light can usually be compensated for by a background correction adjacent to the analyte line.

Users of simultaneous multi-element instruments must verify the absence of spectral interference from an element in a sample for which there is no instrument detection channel. Potential spectral interferences for the recommended wavelengths are given in the table in Attachment 1. The data are intended as rudimentary guides for indicating potential interferences; for this purpose, linear relations between concentration and intensity for the analytes and the interferences can be assumed.

4.1.1 The interference is expressed as analyte concentration equivalents (i.e., false analyte concentrations) arising from 100-mg/L of determined As (at

193,696 ηm) in a sample containing approximately 10-mg/L of Al. According to Attachment 1, 100-mg/L of Al would yield a false signal for As equivalent to approximately 1.3-mg/L. Therefore, the presence of 10-mg/L of A1 would result in a false signal for As equivalent to approximately 0.13-mg/L. The user is cautioned that other instruments may exhibit somewhat different effects and must be evaluated individually since the intensities will vary with operating conditions, power, viewing height, argon flow rate, etc.

4.1.2 The dashes in Attachment 1 indicate that no measurable interferences were observed even at higher interference concentrations. Generally, interferences were discernible if they produced peaks, or background shifts, corresponding to 2 to 5% of the peaks generated by the analyte concentrations.

4.1.3 At present, information on the listed silver and potassium wavelengths is not available, but it has been reported that second-order energy from the magnesium 393.231 ηm wavelength interferes with the listed potassium line at 766.491 ηm.

4.2 Physical Interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, by using a peristaltic pump or by using the standard additions method. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, which affects aerosol flow rate and causes instrumental drift. The problem can be controlled by wetting the argon using a tip washer prior to nebulization or by diluting the sample. Also, it has been reported that better control of the argon flow rate improves instrument performance; this is accomplished with the use of mass flow controllers.

4.3 Chemical Interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, they can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 The Thermo Jarrell Ash ICAP Trace Analyzer uses yttrium as an internal standard and takes into account any interference by ratioing the elements detected against the yttrium detected. All standards, blanks, and samples are spiked with yttrium.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The Chemical Hygiene Plan and MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Inductively coupled argon plasma emission spectrometer

6.1.1 TJA Trace Analyzer

6.1.2 TJA 61E Trace Analyzer

6.**2** Computer-controlled emission spectrometer with background correction

6.2.1 ThermoSPEC Version 6.20

6.**3** Radio frequency generator

6.**4** Argon gas supply: Welding grade or better

6.**5** **8 mL Polypropylene disposable** sample tubes,

6.**6** Automated pipette

6.6.1 Rainin Electronic Digital Pipettes

6.6.2 Eppendorf Research Pipettes

6.**7** **PFA (Teflon®)** screw-top bottles, various sizes

6.8 Plastic Sample Cups, various sizes

6.9 Element DataSystem® LIMS by Promium

7.0 Reagents and Standards

Note: **All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date five years from receipt. All lab prepared regents must be assigned an expiration date one year from preparation.** All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the Low Range Standard (LRS)/low-level continuing calibration verification (LLCCV), at the PQL.

Refer to the Standards/QC Preparation Log for Trace ICP (Attachment 2) for details of all standard preparations. Label each standard bottle with the lot number as described in the log. **All certificates of analysis for vendor stock standards are entered into the LIMS system. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.**

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20th Edition of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

7.2 Concentrated acids – ultra high-purity grade

7.2.1 Hydrochloric acid (HCl), Caledon, Trace Metal Grade

7.2.2 Nitric acid (HNO_3), J.T. Baker, Instra-Analyzed® Reagent, or equipment

7.2.3 The same grade/purity of acids is to be used for all sample preparation, calibration standards, blanks, and QC samples.

7.3 Stock standard solutions – commercially available

7.3.1 The following table contains the stock standards used for ICP-AES analysis. Stock standard expiration dates are provided by the manufacturer.

Manufacturer	Standard
High Purity	SM-770-027 multi-element standard mix (RSTD-1)
High Purity	SM-770-028 part A multi-element standard mix (RSTD-2A)
High Purity	SM-770-028 part B multi-element standard mix (RSTD-2B)
High Purity	SM-770-029 part A multi-element standard mix (RSTD-3A)
High Purity	SM-770-029 part B multi-element standard mix (RSTD-3B)
High Purity	SM-770-030 multi-element standard mix (RSTD-4)
High Purity	SPEX-R multi-element standard mix
High Purity	ICP-AES-CRQL-R multi-element standard mix
Inorganic Ventures	ICV multi-element standard mix
Inorganic Ventures	ICSA multi-element standard mix
Inorganic Ventures	ICSB multi-element standard mix
High Purity	SM-770-0**45 multi-element standard mix (LRS 6010C)**
High Purity	ARSENIC (As) 1,000 PPM single-element standard
High Purity	Internal Mix
High Purity	XCL LCS 10XR multi-element mix
High Purity	XCL 20R multi-element mix

7.4 Intermediate and working standards are prepared at the bench by the analyst as needed. Intermediate and stock standard expiration dates do not exceed the expiration date of the stock standards used.

7.5 Calibration standards are prepared as needed as described on the Standards/QC Preparation Log for Trace ICP.

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, “Receiving Samples” and 4.6, “Storing Samples.” Sample holding times are also listed.

8.2 All samples must be preserved to a pH <2 with HNO_3.

8.3 Samples must be digested and analyzed within 180 days of collection.

8.4 Soil samples must be stored under refrigeration at 4 $^oC \pm 2\ ^oC$. Aqueous samples may be stored at ambient temperature.

8.5 After analysis residual digestate is maintained in the ICP room for three months then returned to the Custodian for storage until disposal into the acid waste stream.

9.0 Quality Control

9.1 Instrument Detection Limit (IDL)

9.1.1 IDLs are determined quarterly for each wavelength used for sample analyses.

9.1.2 Calculate the IDL for each analyte by multiplying by three, the average of the standard deviations calculated from the analysis of seven consecutive standard solutions (containing each analyte in reagent water) on three non-consecutive days (e.g., Monday, Wednesday, and Friday). The concentration of the standard analyzed should be at three to five times the most recently determined IDL values.

9.1.3 The IDL for each analyte must be less than the analyte reporting limit.

9.1.3.1 To meet the requirements of the **DoD**-QSM, the IDL must be less than or equal to the LOD for each analyte.

9.2 Linear Range

9.2.1 The upper limit of the linear range for each ICP-AES instrument needs to be established for each wavelength used, by determining the signal response from a minimum of three, preferably five, different concentration standards across the range and must be verified every six months.

9.2.2 Analyze a high concentration standard, linear range standard (LRS), during a routine analytical sequence. This concentration is the upper limit of the ICP-AES linear range.

9.2.3 The recovery of the LRS must be within ± 10% of the expected value.

9.2.4 Samples with concentrations exceeding the linear range must be diluted.

9.2.5 For North Carolina compliance reporting, the linear range is defined by the highest calibration standard.

9.2.5.1 A high calibration standard that encompasses the concentration of the samples may be used to bracket the sample analyses. This high standard must meet the acceptance criteria in Sections 9.2.3 or 9.2.5.

9.3 Method Blank

9.3.1 A method blank is prepared in every batch of up to 20 samples and is carried through the entire sample preparation and analytical process. This blank is used to ascertain whether sample concentrations reflect contamination.

9.3.2 The method blank must contain absolute values of analyte concentrations below the reporting limit (PQL) or below the applicable action level. If the method blank concentrations exceed the reporting limit, the entire sample batch must be re-prepared along with a new method blank.

9.3.3 To meet the requirements of the **DoD**-QSM, the concentration of the target analytes in the method blank must be ≤ ½ the reporting limit.

9.3.4 For samples submitted for North Carolina compliance reporting, the concentrations of target analytes detected are reported between the MDL and PQL with a "J" flag. Any analytes detected in preparation blanks cannot be > ½ the PQL.

9.4 Matrix Spike

9.4.1 One matrix spike/matrix spike duplicate (MS/MSD) pair or matrix spike/sample duplicate (depending on project requirements) must be prepared with each batch of up 20 samples.

9.4.2 If the sample spikes do not meet the acceptance criteria of 75-125% recovery, the corresponding element is flagged with an "N" on the Form 1 to indicate that the element did not recover in the matrix spike acceptably. A post-digestion spike is performed for those elements that fall outside the limit.

9.4.2.1 To meet the requirements of the **DoD**-QSM, the recoveries of the matrix spike should meet the LCS control limits of 80 – 120%.

9.4.3 The Relative Percent Difference (RPD) between the MS/MSD or sample and sample duplicate must be ≤ 20%. If the criteria is not met, the affected element is flagged with an "*" to indicate poor duplication of results.

9.4.3.1 To meet the requirements of the **DoD**-QSM, report the specific analytes in the original sample as estimated values if the matrix spike and duplicate sample criteria are not met.

9.5 Post Digestion Spike (PDS)

9.5.2.1 Prepare and analyze a PDS, if matrix spike recovery criteria are not met. The spike addition should result in a value that is 2x the RL.

9.5.2.2 To meet the requirements of the DoD-QSM, a PDS must be prepared and analyzed when the serial dilution acceptance criteria are not met or all sample analyte concentrations are less than 50 times the LOD.

9.5.2.3 The PDS results should be within 75% to 125% for Method 6010B and within 80% to 120% for Method 6010C. If not, the data are reported and discussed in the SDG narrative.

9.6 Laboratory Control Sample

9.6.1 A Laboratory Control Sample (LCS) is prepared with every digestion batch of up to 20 samples.

9.6.2 The manufacturer supplies the certified acceptance limits for the solid LCS. A recovery limit of 80 – 120% is required for the aqueous LCS. If the LCS falls outside these control limits, the analysis must be terminated, the problem corrected, and the samples associated with that LCS re-digested and reanalyzed.

9.6.2.1 To meet the requirements of the **DoD**-QSM, the LCS recoveries must be within 80 – 120% of the expected result.

9.7 Serial Dilution

9.7.1 One serial dilution must be performed for each batch.

9.7.2 Prepare a 5x dilution on a sample with an analyte concentration ≥ 50x of the instrument detection limit in the original sample. The results of the dilution must then agree within ± 10% of the original sample results. If the dilution does not met this criteria, then flag the appropriate elements with an "E" to indicate that interference exists. The "E" flag is not used for **DoD**-QSM analyses. Refer to the **DoD**-QSM "J" flag.

9.7.2.1 To meet the requirements of the **DoD**-QSM, a PDS must be prepared and analyzed when the serial dilution acceptance criteria are not met or all sample analyte concentrations are less than 50 times the LOD.

9.8 Field and/or Equipment Blanks

9.8.1 Samples identified as field and/or equipment blanks should not be used for sample spike, duplicate, or serial dilution analysis. They are supplied to the laboratory at the discretion of the client.

9.9 Contingency

9.9.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.9.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.9.3 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 <u>Calibration and Standardization</u>

10.1 For Method 6010C, a lower limit of quantitation check (LLQC) is analyzed after the PQL is determined and on an as-needed basis to demonstrate the desired detection capability. The LLQC and the Low Range Standard (LRS) are prepared at the same concentration. The only difference is that the LLQC is carried through

the entire sample preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC are detected within ± 30% of their true values.

10.1.1 The LRS, for Method 6010B, is a standard at the RL, with advisory acceptance criteria of ± 50%.

10.1.2 To meet the requirements of the **DoD**-QSM, the recovery of each analyte in the LRS **6010C** must fall within ± 20% of the expected value.

10.2 The instrument calibration standards are analyzed at the beginning of the analytical sequence and entered on the Trace ICP run log (Attachment 3). Calibration is further discussed in the Procedure section that follows.

10.3 Initial Calibration

10.3.1 The initial calibration consists of a zero concentration standard or calibration blank (ICB) and **one** calibration standard. The analytes are divided into the following four standard solutions and analyte concentrations:

Standard Name	**Analyte – concentration in µg/L**
RSTD-1	Cd, Cr, Cu, Pb, Ni, Se, Tl, and Zn = 2,000; Ag = 200,
RSTD-2	As, Ba, Be, Co, Mn, Mo, and V = 2,000; Fe = 50,000
RSTD-3	K, Na, Al, Mg, and Ca = 100,000
RSTD-4	Sn, Bi, Ti, Sb, and B = 2, 000

10.3.2 The calibration blank consists of 5% HCl and 1% HNO_3 in reagent water.

10.3.2.1 A calibration blank is analyzed as part of both the initial calibration (ICB) and continuing calibration (CCB).

10.4 Initial Calibration Verification (**LLICV**/ICV)

10.4.1 The ICV consists of second source mid-level calibration standard.

10.4.**1.1** The **Inorganic Ventures** solution**s are** used to prepare the ICV and contain all of the standards in the table in section 10.3.

10.4.**1.2** The results of the ICV must agree within ± 10% of the true value for each component. If this criterion is not met, terminate the run and re-calibrate the instrument. If the re-calibration does not

produce acceptable ICV results, shut down the instrument and perform instrument maintenance.

10.4.2 For Method 6010C, a low-level initial calibration verification (LLICV) standard is also prepared, at the RL, and is from the same source as the calibration standard.

10.4.**2.1** The acceptance criterion for the LLICV is ± 30% of its true **value**. **If this criterion is not met, terminate the run and re-calibrate the instrument. If the re-calibration does not produce acceptable ICV results, shut down the instrument and perform instrument maintenance.**

10.5 Initial Calibration Blank (ICB)

10.**5**.**1** Follow the **LL**ICV/ICV with the ICB. The absolute value of the blank must be less than the laboratory reporting limit (PQL). If not, repeat the blank analysis. If still above the reporting limit, terminate the analysis and recalibrate. If re-calibration does not produce acceptable ICB results, shut down the instrument and perform instrument maintenance.

10.**5**.**1**.1 To meet the requirements of the **DoD-**QSM, the analyte concentration in the ICB must be ≤ LOD.

10.5.1.2 For North Carolina compliance reporting, the concentrations of target analytes detected are reported between the MDL and PQL with a "J" flag. Any analytes detected in ICB cannot be > ½ the PQL.

10.**6** Continuing Calibration Verification (**LLCCV**/CCV)

10.**6**.1 For Method 6010C, a low-level continuing calibration verification (LLCCV) standard is also prepared, at the RL, and is from the same source as the calibration standard. It is analyzed every 19 samples and at the end of the analytical run.

10.6.1.2 The acceptance criterion for the LLCCV is ± 30% of its true value.

10.**6**.2 Verify the calibration **using a Continuing Calibration Verification (CCV) standard** every 10 samples and at the end of analytical run. **The CCV is from the same source as the calibration standard and the**

concentrations of the analytes is roughly one-half of the initial calibration standard.

10.**6**.**2.1** The results of the CCV must agree within ± 10% of the expected value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

10.7 Continuing Calibration Blank (CCB)

10.**7.1** A CCB must be analyzed at each wavelength used for analysis immediately after every continuing calibration **verification** standard.

10.**7.2** The acceptance criteria stated in Sections 10.4.4**,** 10.4.4.1**, and 10.4.4.2** also apply to the CCB.

10.**8** Interference Check Sample (ICS)

10.8.1 The ICS consists of the ICSA and ICSAB solutions. Analyze the ICSA and ICSAB consecutively at the beginning the analytical run.

10.8.2 The results of the ICS must agree within ± 20% of the accepted values or 2X the RL, whichever is greater. If not terminate the analysis and recalibrate the instrument. If re-calibration does not fix the problem, shut down the instrument and perform instrument maintenance.

10.**8**.2.1 To meet the requirements of the **DoD**-QSM, the absolute value of the concentration of all the non-spiked analytes in the ICS-A must be < LOD (unless the analyte is a verified trace impurity from one of the spiked analytes).

11.0 <u>Procedure</u>

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 TJA Trace Analyzer Set Up and Operating Procedure

11.1.1 Cooling Water

11.1.1.1 Turn on the recirculation pump. Thirty (30) psig is required at a flow rate of 700 mL/min.

11.1.2 Argon Supply

11.1.2.1 Open the argon supply to the instrument.

11.1.2.2 A volume rate of 30 SCFH and delivery pressure of 60 psig is required.

11.1.3 Venting System

11.1.3.1 A permanent vent has been installed to provide a proper draft in the torch compartment. A torch fan is permanently installed to force a 22 mph-draft across the end of the plasma torch.

11.1.4 Water in Drain Barrel

11.1.4.1 A hose is connected to the drain outlet from the spray chamber to allow the flow of unused sample waste from the chamber. This drain line acts as a positive seal to the spray chamber; therefore, liquid must always be present in the drain line.

11.2 Power Up (TJA Trace Analyzer)

11.2.1 To start the ICP Trace Analyzer, turn on the video display, the printer, the computer, and the autosampler.

11.2.2 At the "C" prompt, Type "P" and press “ENTER.” This will start the Thermospec™ software and bring up the main menu.

11.2.3 To start the RF generator and the plasma torch, select “SETUP.” Then select “PLASMA CONTROL PANEL” and press “ENTER.” Select “F1.”

11.2.4 The default startup time, during which argon purges the spray chamber and the plasma torch, is 90 seconds. The purge time can be reduced to as little as 20 seconds by pressing “PURGE TIME” if the plasma has been off for less than 15 minutes.

11.2.5 After the purge time and power have been set, the automated plasma startup sequence is initiated by pressing “CONTINUE.”

11.2.6 If the pump and gas have not been started, select “F2” and start the pump rate at 99 RPM and switch the nebulizer gas to “ON.”

11.2.7 The total flush time and rinse time between samples is set at 60 seconds.

11.2.8 Press "LEVELS" if you want to change any of the plasma operating conditions.

11.2.9 The plasma torch should be allowed to run for at least 30 minutes after ignition to reach optimum stability.

11.2 Analysis

11.2.1 Profile

11.2.1.1 Profiling the instrument assures that the optical pathway is clean and in alignment. Emission intensities are essential to accurate data.

11.2.1.2 At the THERMOSPEC main menu, select setup and highlight profile then enter.

11.2.1.3 Begin aspirating 1 ppm As standard for analysis.

11.2.1.4 Press F3 to Automatic Profile. When the sample is sufficiently saturating the spray chamber and torch (60 seconds), press "F1", "RUN".

11.2.1.4.1 At the end of the integration, a peak graph will be displayed. Note the intensity reading. This will indicate a need to clean the optical path or adjust the alignment.

11.2.1.4.2 Also note the peak position on the graph. This value should be between ± 0.2. If it is not, the spectrum shifter must be adjusted, and the profile rerun. Continue this process until the peak position is acceptable.

11.2.1.5 Discontinue the arsenic when profiling is complete. Exit profile to the main menu.

11.3 Autosampler Table Setup

11.3.1 Obtain all data necessary from each case that will be set up on the run, i.e. preparation logs, log-in chain of custody reports from the LIMS. The information contained on these sheets will be used to create the autosampler table.

11.3.2 From the main menu of Thermospec, select "OPERATION," and then "Autosampler Setup," then press enter. The software will prompt for an autosampler table name. An existing table can be edited from this point, or a new autosampler table name can be typed in at this point. Press enter. Press F3 "Add Set." Enter the appropriate run parameters to be used. Parameters that should always be entered here are Method Name, Rinse Time (60 seconds), # of Unknowns in this set, Default Limit Check Table Name (Sample). Press the F1 "EdSamples" key. This will allow for sample IDs to be entered.

11.3.3 Under the column titled "Sample Name," enter the CompuChem sample ID. Be certain the proper check table is selected for each analysis that is assigned. The "F" key allow for various modifications to the autosampler table. Use these keys as necessary. It is very important that the proper QC samples be inserted into the table at the necessary frequencies. See **Section** 9.0 for a description of necessary QC and frequencies.

11.3.4 Also, if the "Alt" key on the keyboard is pressed, additional options will appear. One of these options is "EdSampInfo." This option is selected by pressing the "Alt" key and the "F2" key simultaneously. From this screen, additional comments can be added that will appear on the raw data printout. Under the column titled "Comment," the SDG, client sample ID and dilution of the sample should be entered.

11.3.5 When all entries are finished, press the "F9," Done/Keep key to save the table. It is important to remember the name of the table created, as this will be used to start the analysis.

11.3.6 Create a configuration file to store all analytical data. From the main menu of THERMOSPEC select "SETUP" and highlight "CONFIGURATION." Press "Enter" twice then "F9" four times. This will provide the screen to enter the configuration file name. Configuration file names should identify the analyst and the date of the analysis, as well as the instrument being used (P3 or P4). Use the following scheme (iimmdd) to construct the file name. The letter "i" for initials, the "m" for the month, and the letter "d" for the day. If there are more runs in a day, letters of the alphabet are used as a suffix. In this example: P3JC0922B,

identifies a third run on September 22 by analyst JC on instrument P3. JUNK files should be used between analyses to prevent files from being corrupted with other data.

11.3.7 Proceed to analysis. From the main menu of THERMOSPEC select "OPERATION." Highlight "ANALYSIS" then "Enter." Confirm that the correct method is being used and select the autosampler table to be used. Continue to follow on-screen instructions to initiate the run.

IMPORTANT: If the run should fail at any time, remember to change the configuration file name before restarting the analytical run.

11.4 Standardization and Analysis

11.4.1 Start autosampler table and follow the instructions.

11.4.1.1 Select "Operation" and highlight "Analysis."

11.4.1.2 Enter the correct method of analysis and select the autosampler table for samples to be analyzed.

11.4.2 Solid samples and liquid samples have different matrices due to sample preparation procedures. Calibration standards and quality control standards should be prepared to match the matrix of the samples being analyzed. While constructing autosampler tables, give consideration to the matrix of the samples.

11.4.3 Fill all standards cups and QC cups with the appropriate solutions and enter the information on the Trace ICP Run Log (Attachment 3.) The autosampler table provides the information for cup positions. Standards cups positioning may change depending on the number of samples.

Caution: Accuracy of the sequence setup is extremely important. Improper cup positioning will seriously affect analysis to the point that there may not be any salvageable data.

11.4.4 Press "F1" to begin the analytical run.

Caution: Sample racks should be loaded accurately following the rows and positions provided by the autosampler table. Samples in

the wrong position can produce erroneous data that may not be detectable.

11.4.5 Although, software drives the instrument operation, if possible the instrument should not be left completely unattended. In the course of the run, matrix spike analyses must be evaluated to determine percent recoveries. Some samples will have concentrations of analytes that are beyond the established linear ranges and will need to be diluted and reanalyzed within those ranges.

For samples submitted for North Carolina compliance reporting, dilutions and re-analyses are required whenever target analytes exceed the highest calibration standard. Serial dilution analyses should be evaluated for proper dilution. Responding to the need for dilutions or a PDS analysis quickly within the same analytical run saves time by not requiring an additional run and improves productivity.

11.4.6 Attention should be given to the QC analyses. Analytes may be found to be outside of their established control limits. The loss of one or more analytes does not necessarily mean the run has ended. Early detection of failed analytes by the analyst will prevent lost time and productivity.

11.4.7 Standards cups have limited capacity and should be filled frequently and monitored at all times.

11.4.8 The analytical run is not complete until all final paperwork is complete. The run log must be completed by the analyst and the QC/Standards Preparation log completed for the day, as well as completing the internal chain of custody form (Attachment 4).

11.5 Transferring Data

11.5.1 At the completion of the analytical run, exit analysis and return to the THERMOSPEC main menu. Change the configuration file name.

11.5.2 Exit THERMOSPEC and return to Desktop. Double-click on the Explorer icon. While in Explorer, open the STATION directory in the left column, and then open the BIN directory. In the right column, find the name of the file from the analytical run that was just finished. Next, find the M**arrs on Marley** directory in the left column and open that directory. Click on the file name in the right column and copy it to the appropriate directory in

M**arrs on Marley** (P3 ICP Trace Files or P4 ICP Trace Files.) Explorer can then be closed.

11.5.3 Transfer the file to the computer used to run the ICP Checker program. The ICP Checker function combines two pages of data onto one page. The run log is also generated at this time.

11.6 Instrument Shutdown

11.6.1 TJA Trace Analyzer (P3 or P4)

11.6.1.1 Aspirate reagent water for approximately one minute.

11.6.1.2 From the MAIN MENU go to SETUP and PLASMA CONTROL PANEL and press enter.

11.6.1.3 Press F7 for PLASMA SHUT off.

11.6.1.4 Remove pump tubing from peristaltic pumps.

12.0 <u>Data Analysis and Calculations</u>

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$Standard\ deviation = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.3.2 Matrix spikes:

$$\%\ R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.4 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{(Value\ 1\ +\ Value\ 2)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value\ -\ \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Calculating Dilutions

12.7.1 If a sample concentration exceeds the linear range of the instrument, a dilution reanalysis must be performed. For samples submitted for **DoD**-QSM and North Carolina compliance reporting, dilutions and re-analyses are required whenever target analytes exceed the highest calibration standard.

12.7.2 Determine a level of dilution that will result in a value within the upper half of the linear range or, for samples submitted for **DoD**-QSM and North Carolina compliance reporting, within the upper half of the calibration range. This is an acceptable dilution.

12.7.3 A 10x dilution is performed using 1-mL sample plus 9-mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

12.8 Concentration

12.8.1 All results for aqueous samples are reported in μg/L as follows.

$$\text{Concentration } (\mu g/L) = \frac{C \times V \times D}{W}$$

Where: C = Concentration (μg/L) from curve
V = Final volume of digestate (L)
W = Volume of sample (L)
D = Dilution factor

12.8.2 All results for solid samples are reported in mg/Kg as follows.

12.8.2.1 A separate determination of percent solids must be performed.

12.8.2.2 The concentration determined in the digestate is to be reported on the basis of the dry weight of the sample.

$$\text{Concentration (mg/Kg)} = \frac{C * V}{W * S}$$

Where: C = Concentration (μg/L)
V = Final volume of digestate (L)
W = Weight of wet sample (gm)
S = % Solid / 100
D = Dilution factor

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits, instrument detection limits, and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or

eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal" requiring laboratory procedures for recycling waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96, Method 6010B, Update IV, February 2007, Method 6010C, Revision 3

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 **TNI** Standards, effective July **2011**, plus revisions

16.7 EPA QA/G6: Guidance for the Preparing of Standard Operating Procedures (SOPs) EPA/600/B-07/001, April **2007**.

16.8 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.9 Sample Control SOP 4.6, "Storing Samples"

16.10 TJA Trace Analyzer Set-up and Operating Manual

16.11 Department of Defense Quality Systems Manual of Environmental Laboratories, Version 4.**2**, **October 25, 2010**

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 – Analyte Concentration Equivalents Arising from Interference at the 100 mg/L Level

17.2 Attachment 2 – Example Daily Standards/QC Preparation for Trace ICP (P3&P4)

17.3 Attachment 3 – Example Trace ICP Run Log (P3)

17.4 Attachment 4 – Example Internal Chain of Custody for Metals Analysis

17.5 Attachment 5 – Target Analytes

Attachment 1

Analyte Concentration Equivalents Arising from Interference at the 100-mg/L Level

Analyte	Wavelength (ηm)	Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Tl	V
Aluminum	308.215	--	--	--	--	--	0.21	--	--	_--	1.4
Antimony	206.833	0.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Arsenic	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Barium	455.403	--	--	--	--	--	--	--	--	--	--
Beryllium	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Boron	249.773	0.04	--	--	--	0.32	--	--	--	--	--
Cadmium	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Calcium	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Chromium	267.716	--	--	--	--	0.003	--	0.004	--	--	0.04
Cobalt	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Copper	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Iron	259.940	--	--	--	--	--	0.12	--	--	--	--
Lead	220.353	0.17	--	--	--	--	--	--	--	--	--
Magnesium	231.604	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Manganese	196.026	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Molybedenum	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Nickel	279.079	--	--	--	--		--	--	--	--	--
Selenium	257.610	0.23	--	--	--	0.09	--	--	--	--	--
Silicon	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Sodium	588.995	--	--		--	--	--	--	--	0.08	--
Thallium	190.864	0.30	--		--	--	--	--	--	--	--
Vanadium	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zinc	213.856	--	--	--	0.14	--	--	--	0.29	--	--

[a] Dashes indicate that no interference was observed even when interferents were introduced at the following levels:

Al, Ca, Mg - 1000 mg/L; Ca, Fe - 1000 mg/L; Cr, Cu, Mn, Tl, V - 200 mg/L

[b] The figures recorded as analyte concentrations are not the actual observed concentrations; to obtain those figures, add the listed concentration to the interferent figure.

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Attachment 2

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 1 of 3 CompuChem a division of Liberty Analytical Corp. **Worksheet 1 P(6) R5**

STANDARDS and REAGENTS TRACEABILITY Seq. File Name:_____

	Ref #*	Manufacturer	Lot #	Expiration Date
HNO_3 [Baker Instra-analyzed Trace-Metals grade]		JT Baker		
HCl [Baker Instra-analyzed Trace-Metals grade]		Caledon		
SM-770-027 multi-element standard mix (RSTD-1)	1F10007	High Purity	1115721	06/08/12
SM-770-028 part A multi-element standard mix (RSTD-2A)	0J18002	High Purity	1028535	10/13/11
SM-770-028 part B multi-element standard mix (RSTD-2B)	0J18001	High Purity	1028536	10/13/11
SM-770-029 part A multi-element standard mix (RSTD-3A)	1D12001	High Purity	0805135	04/08/12
SM-770-029 part B multi-element standard mix (RSTD-3B)	0G31002	High Purity	1010601	01/27/12
SM-770-030 multi-element standard mix (RSTD-4)	1F10008	High Purity	1115724	06/08/12
SPEX-R multi-element standard mix	1F10006	High Purity	1115722	06/08/12
ICP-AES-CRQL multi-element standard mix	1B16011	High Purity	1031509	11/12/11
ICCV-3 multi-element standard mix	1J06006	Inorganic Venture	E2-MEB395052	11/01/12
ICCV-4 multi-element standard mix	1J06007	Inorganic Venture	E2-MEB395053	11/01/12
ICSA multi-element standard mix	1F06001	Inorganic Venture	D2-MEB348015	06/01/12
ICSB multi-element standard mix	1F06002	Inorganic Venture	D2-MEB346026	06/10/12
ARSENIC (As) 1,000 PPM single-element standard	0L01020	High Purity	1021405	06/01/12
SM-770-020 Internal Standard Mix	1D21013	High Purity	1110812	04/20/12
SM-770-040 multi-element CCV mix (Soln A)	1D21005	High Purity	1110810	04/20/12
SM-770-040 multi-element CCV mix (Soln B)	1D21005	High Purity	1110811	04/20/12
SM-770-045 LRS 6010C (Soln A)	1B16001	High Purity	1101110	01/12/12
SM-770-045 LRS 6010C (Soln B)	1B16002	High Purity	1101111	01/12/12

Multi-Element Stock Standard Concentrations:

Stock Standard	Analyte Concentrations (µg/mL)
RSTD-1 multi-element standard mix	Cd:1,000 Cr:1,000 Cu:1,000 Pb:1,000 Ni:1,000 Se:1,000 Ag:100 Tl:1,000 Zn:1,000
RSTD-2A/2B multi-element standard mix	Part A: As:1,000 Ba:1,000 Be:1,000 Co:1,000 Mn:1,000 Mo:1,000 V:1,000 Part B: Fe:25,000
RSTD-3A/3B multi-element standard mix	Part A: Ca:10,000 Mg:10,000 K:10,000 Na:10,000 Part B: Al:10,000
RSTD-4 multi-element standard mix	Sb:1,000 Bi:1,000 B:1,000 Sn:1,000 Ti:1,000
SPEX-R multi-element standard mix	Mo:1,000 Sn:1,000 Bi:1,000 Tl:1,000 B:1,000
ICP-AES-CRQL-R multi-element standard mix	Ag:10 Be:5 Cd:5 Co:50 Cr:10 Cu:25 Mn:15 Ni:40 Tl:25 V:50 Zn:60 Pb:10 Se:35 As:10 Sb:60
LRS 6010C multi-element standard mix	Al:200 Ba:200 K:5000 Mg:5000 Na:5000 Ca:5000 Ag:5 Be:5 Cd:5 Co:20 Cr:10 Cu:5 Fe:200 Mn:10 Ni:10 Tl:30 V:20 Zn:30 Pb:10 Se:5 As:10 Mo:50 Sn:50 Bi:20 Ti:50 Sb:30 B:50
SM-770-020 Internal Standard Mix	Li:50,000 Y:250
IICCV-3 multi-element standard mix	Ca: 1,100 K:1,100 Na:1,100 Mg:600 Fe:510 Al:260 As:110 B:110 Bi:110 Pb:110 Se:110 Tl:110 Zn:110 Ag:55 Ba:55 Cd:55 Co:55 Cr:55 Cu:55 Mn:55 Ni:55 V:55
IICCV-4 multi-element standard mix	Mo:110 Sb:110 Sn:110 Ti:110
IICSA multi-element standard mix	Al:5,000 Ca:5,000 Mg:5,000 Fe:2,000
IICSB multi-element standard mix	Cd:100 Ni:100 Zn:100 Sb:60 Ba:50 Be:50 Co:50 Cr:50 Cu:50 Mn:50 V:50 Ag:20 As:10 Tl:10 Pb:5 Se:5

Entered By: _____

Reviewed By: _____ Date: _____

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log 10/17/11: jcf/v

**High Purity is abbreviated "HP" on reporting forms

Note: This page subject to change without notice.

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Attachment 2 (continued)

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 2 of 3 CompuChem a division of Liberty Analytical Corp. **Worksheet 1 P(6) R5**

Standard Lot Number*	LIMS ID Number	Preparation instructions	Resultant Analyte Conc. (µg/L)
S0/ICB/CCB–		Place in a 500 mL volumetric flask: 100 mL deionized water, 20 mL concentrated HNO_3, and 100 mL concentrated HCl. Bring up to volume with deionized water. Transfer to a 2L Teflon bottle and add 1.5L deionized water. Final volume 2 L. Prepare as needed.	N/A
RSTD-1–		In a 200 mL volumetric flask, combine 150 mL deionized water, 2 mL concentrated HNO_3, and 10 mL concentrated HCl. Pipet 0.4 mL RSTD-1 into flask. Bring up to 200 mL volume with deionized water. Prepare as needed.	Cd:2,000 Cr:2,000 Cu:2,000 Pb:2,000 Ni:2,000 Se:2,000 Ag:200 Tl:2,000 Zn:2,000
RSTD-2–		In a 200 mL volumetric flask, combine 150 mL deionized water, 2 mL concentrated HNO_3, and 10 mL concentrated HCl. Pipet 0.4 mL each RSTD-2A and RSTD-2B into flask. Bring up to 200 mL volume with deionized water. Prepare as needed.	As:2,000 Ba:2,000 Be:2,000 Co:2,000 Fe:50,000 Mn:2,000 Mo:2,000 V:2,000
RSTD-3–		In a 200 mL volumetric flask, combine 150 mL deionized water, 2 mL concentrated HNO_3, and 10 mL concentrated HCl. Pipet 2.0 mL each RSTD-3A and RSTD-3B into flask. Bring up to 200 mL volume with deionized water. Prepare as needed.	K:100,000 Na:100,000 Al:100,000 Mg:100,000 Ca:100,000
RSTD-4–		In a 200 mL volumetric flask, combine 150 mL deionized water, 2 mL concentrated HNO_3, and 10 mL concentrated HCl. Pipet 0.4 mL RSTD-4 into flask. Bring up to 200 mL volume with deionized water. Prepare as needed.	Sn:2,000 Bi:2,000 Ti:2,000 Sb:2,000 B:2,000
ICV1–		Place in a 500 mL volumetric flask: 300 mL deionized water, 5 mL concentrated HNO_3 and 25 mL concentrated HCl. Pipet 5.0 mL ICCV-3 into flask and 5.0 mL ICCV-4. Bring up to volume with deionized water. Transfer to a 500mL Teflon bottle. Final volume 500 mL. Prepare as needed.	Al:2,600 Ba:550 K:11,000 Mg:6,100 Na:11,000 Ca:11,000 Ag:550 Be:550 Cd:550 Co:550 Cr:550 Cu:550 Fe:5,100 Mn:550 Ni:550 Tl:1,100 V:550 Zn:1,100 Pb:1100 Se:1,100 As:1,100 Sb:1,100 Mo:1,100 Sn:1,100 Bi:1,100 Ti:1,100 B:1,100
CCV–		Place in a 500-mL volumetric flask: 300 mL deionized water, 20 mL concentrated HNO_3 and 100 mL concentrated HCl. Add 50 ml of SM-770-40 Soln A and Soln B. Bring up to volume with deionized water. Transfer to a 2000mL Teflon bottle and add 1.5 L of deionized water. Prepare as needed.	Al:50,000 Ba:1,000 K:50,000 Mg:50,000 Na:50,000 Ca:50,000 Ag:100 Be:1,000 Cd:1,000 Co:1,000 Cr:1,000 Cu:1,000 Fe:25,000 Mn:1,000 Ni:1,000 Tl:1,000 V:1,000 Zn:1,100 Pb:1,000 Se:1,000 As:1,000 Mo:1,000 Sn:1,000 Bi:1,000 Ti:1,000 Sb:1,000 B:1,000
LLICV/LLCCV/LRS 6010C-		In a 500 mL volumetric flask, combine 200 mL deionized water, 5 mL concentrated HNO_3 and 25 mL concentrated HCl. Pipet 0.5 mL of each LRS 6010C Soln A and Soln B. Bring up to volume with deionized water and transfer to a 500mL Teflon bottle. Final volume 500 mL. Prepare as needed.	Al:200 Ba:200 K:5000 Mg:5000 Na:5000 Ca:5000 Ag:5 Be:5 Cd:5 Co:20 Cr:10 Cu:5 Fe:200 Mn:10 Ni:10 Tl:30 V:20 Zn:30 Pb:10 Se:5 As:10 Mo:50 Sn:50 Bi:20 Ti:50 Sb:30 B:50

Standard Solutions Prepared By: _____

Entered By: _____

Reviewed By: _____ Date: _____

*Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

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Attachment 2 (continued)

Daily Standards/QC Preparation Log for Trace ICP (P3 & P4) 3 of 3 CompuChem a division of Liberty Analytical Corp. **Worksheet 1 P(6) R5**

Standard Lot Number*	LIMS ID Number	Preparation instructions	Resultant Analyte Conc. (µg/L)
As–		In a 100 mL volumetric flask, combine 50 mL deionized water, 1 mL concentrated HNO_3, 5 mL concentrated HCl, and 0.1 mL 1000-ppm As standard. Bring to final 100 mL volume	As:1,000
ICSA–		In a 500-mL volumetric flask, combine 350 mL deionized water, 5 mL concentrated HNO_3, and 25 mL concentrated HCl. Pipet 25.0 mL ICSA into flask. Bring up to volume with deionized water and transfer to a 500mL Teflon bottle. Final volume 500 mL.	Al:250,000 Ca:250,000 Fe:100,000 Mg:250,000
ICSAB–		In a 500-mL volumetric flask, combine 350 mL deionized water, 5 mL concentrated HNO_3, and 25 mL concentrated HCl. Pipet 25.0 mL ICSA and 5.0 mL ICSB and 0.5 mL of Compuchem SPEX into flask. Bring up to volume with deionized water and transfer to a 500mL Teflon bottle. Final volume 500 mL.	Ag:200 Al:250,000 Ba:500 Be:500 Ca:250,000 Cd:1000 Co:500 Cr:500 Cu:50 Fe:100,000 Mg:250,000 Mn:500 Ni:1000 Tl:100 V:500 Zn:1000 Pb:60 Se:50 Sb:600 As:100 Sn:1,000 Bi:1,000 Mo:1,000 Ti:1,000 B:1,000
EPA-CRI-		In a 500 mL volumetric flask, combine 300 mL deionized water, 5 mL concentrated HNO_3, and 25 mL concentrated HCl. Pipet 0.50 mL ICP-AES-CRQL-R and 25 µL Compuchem SPEX-R into flask. Bring up to volume with deionized water and transfer to a 500mL Teflon bottle. Final volume 500 mL. Prepare as needed.	Ag:10 Be:5 Cd:5 Co:50 Cr:10 Cu:25 Mn:15 Ni:40 Tl:25 V:50 Zn:60 Pb:10 Se:35 As:10 Sb:60 Sn:50 B:50 Mo:50 Bi:50 Ti:50
P3RINSE–		In a 10 L container, combine 8 L deionized water and 400 mL concentrated HNO_3 Fill with deionized water. Final volume 10 L.	N/A
P4RINSE–		In a 10 L container, combine 8 L deionized water and 400 mL concentrated HNO_3 Fill with deionized water. Final volume 10 L.	N/A
P5RINSE–		In a 10 L container, combine 8 L deionized water and 400 mL concentrated HNO_3 Fill with deionized water. Final volume 10 L.	N/A
P3INTSTD–		In a 10 L container, combine 8 L deionized water, 400 mL concentrated HNO_3, and 200 mL Internal Mix. Fill with deionized water. Final volume 10 L.	Li:1,000,000 Y:5,000
P4INTSTD–		In a 10 L container, combine 8 L deionized water, 400 mL concentrated HNO_3, and 200 mL Internal Mix. Fill with deionized water. Final volume 10 L.	Li:1,000,000 Y:5,000
P5INTSTD–		In a 10 L container, combine 8 L deionized water, 400 mL concentrated HNO_3, and 200 mL Internal Mix. Fill with deionized water. Final volume 10 L.	Li:1,000,000 Y:5,000

Standard Solutions Prepared By: _____

Entered By: _____

Reviewed By: _____ Date: _____

*Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

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Attachment 3

Daily Instrument **Maintenance:** _____

Nebulizer pressure: _____
Peak position: _____
Peak intensity: _____
Vernier position: _____

Printed:

Method(s (circle **ILM05.4** **6010B** **6010C** **Other** _____

Operator(s) _____ **Date/Time** _____ **to** _____

↑	Specified element(s) fail(s) high
↓	Specified element(s) fail(s) low
RR	Re-run sample(s) neat
RR #X	Re-run at a #-fold dilution
#X	Sample run at a #-fold dilution (aliquot of sample diluted with acidified water)
L	Matrix = leachate
W	Matrix = water
S	Matrix = solid
B	Matrix = biological tissue

SDG	Matrix	Comments
1001011		
1001015		

ICP Run Log

File Name:

Sample ID	Client ID	Sample	DF	Date	Time	SDG	Action Needed

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Reviewed By: _____ Date: _____ Page 1 of 1

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Attachment 4

INTERNAL DIGESTION COC

Printed: 5/28/2010 2:13:28PM

Lab Number	Client ID	Sample Type	

Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date
Relinquished By	Date	Received By	Date

Page 1 of 1

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Attachment 5

Analyte	
Aluminum	Al
Antimony	Sb
Arsenic	As
Barium	Ba
Beryllium	Be
Cadmium	Cd
Calcium	Ca
Chromium	Cr
Cobalt	Co
Copper	Cu
Iron	Fe
Lead	Pb
Magnesium	Mg
Manganese	Mn
Nickel	Ni
Potassium	K
Selenium	Se
Silver	Ag
Sodium	Na
Thallium	Tl
Vanadium	V
Zinc	Zn
Bismuth	Bi
Molybdenum	Mo
Tin	Sn
Titanium	Ti
Boron	B

SOP 3.2.1.9

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP316 SOP Section #: 3.2.1.9 Revision #: 6

SOP Title: Determination of Metals by Inductively Coupled Plasma-Mass Spectrometry (ICP-MS) by SW-846 Method 6020/6020A

Effective date: (QA fills in) 10/18/11

- Procedure prepared by: Judith Dozier Date: 10/18/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [signature] Date: 10/18/11
- Reason for change: Annual review; addition of CIMS information
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Ed., Update III 12/96 method 6020 and Update IV 2/07, method 6020A; DoD-QSM version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 316: Determination of Metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) by SW-846 Method 6020/6020A

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Instrument Procedure 316: Determination of Metals by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) by SW-846 Method 6020/6020A

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the determination of trace concentration of elements in water samples and in waste extracts or digests. When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial wastes, soil, sludge, sediment and other solid wastes for which total (acid-leachable) elements are required.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into a mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratio and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate problems. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

3.0 Definitions

3.1 Dissolved metals – metals that will pass through a 0.45 micron membrane filter.

3.2 Total metals – the concentration of metals determined on an unfiltered liquid sample following vigorous digestion.

3.3 Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte **(40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.4 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.5 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL $<$ LOD $\leq$ LOQ.

3.6 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.7 Reporting Limit (RL) –

3.7.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration.

3.7.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported between the DL and the LOQ must be flagged as estimated values.

3.8 Reporting Units – µg/L for water, mg/Kg for soils

3.9 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples (excluding PE samples) received within a case, or

- each 7 calendar day period during which field samples in a case are received beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.10 m/z = mass to charge ratio

3.11 Instrument Detection Limits (IDL) - determined by multiplying by three the standard deviation obtained for the analysis of a standard solution (each analyte in reagent water) at a concentration of 3x to 5x IDL on three nonconsecutive days with seven consecutive measurements per day.

3.12 **DoD**-QSM – Department of Defense Quality Systems Manual

3.13 LIMS – Laboratory Information Management System

3.14 Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

4.0 Interferences

4.1 Isobaric elemental interferences result from isotopes of different elements forming singly or double charged ions of the same nominal mass-to-charge ratio that cannot be resolved by the mass spectrometer. All elements determined by this method have at least one isotope free of isobaric elemental interference. Only selenium-82, of the analytical isotopes recommended, has an isobaric elemental interference. If greater sensitivity is desired, alternative analytical isotopes with higher natural abundances can be selected, but an isobaric elemental interference may occur. When this happens, all data obtained must be corrected by measuring the signal from another isotope of the interfering element and subtracting the appropriate signal ratio from the isotope of interest. Records need to be maintained of this correction process and should be included in the submitted data package.

4.2 Physical interferences are generally considered to be effects associated with the ICP analysis, especially with samples containing high dissolved solids concentrations. Deposits can form on the extraction and/or skimmer cones, reducing the effective diameter and, therefore, the ion transmission. Physical interferences may occur in the transfer of solution to the nebulizer, at the point of aerosol formation and transport to the plasma, or during the excitation and ionization processes within the plasma.

4.3 Abundance sensitivity relates to the degree the wings of a mass peak contribute to adjacent masses. It is affected by ion energy and mass filter operating pressure. The wing overlap interference may result when a small ion peak is being measured adjacent to a large peak. When this occurs, the resolution of the spectrometer should be adjusted to minimize the condition.

4.4 Isobaric polyatomic ion interferences are caused by ions comprised of more than one atom that have the same nominal mass-to-charge ratio as the isotope of interest and cannot be resolved by the spectrometer. The ions are generally formed in the plasma or interface system from support gases or sample components. These interferences must be recognized and appropriate correction made to the data unless they can be avoided by selecting alternative analytical isotopes.

4.5 Memory interferences occur when isotopes of elements in a previous sample contribute to the signals measured in a new sample. These memory effects (carry over) may develop from sample deposition on the sampler and skimmer cones or from the buildup of sample material in the plasma torch and spray chamber. Flushing the system with a rinse blank between samples can minimize the effect. This problem should be recognized during an analytical run and suitable rinse time used to eliminate it.

By analyzing a standard containing the elements at 10X the upper end of the linear range and following that with the analysis of the rinse blank at designated intervals, the analyst should be able to estimate suitable rinse times. From the length of time necessary to reduce analyte signals to within a factor of 10 of the MDL, the analyst has the necessary rinse time. The assessment of memory interferences can also take place by using a minimum of three replicate integrations for data acquisition. If the integrated signal value drops drop consecutively, this may be a sign to the analyst of a memory effect and that should direct the analyst to evaluate the analyte concentration in the previous sample to determine if it had a high value. If suspected, the sample should be reanalyzed after a long rinse period.

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5.0 Safety

5.1 Many metal salts are extremely toxic if inhaled or swallowed, so wash your hands after handling salts.

5.2 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.3 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for solvents and reagents used in the laboratory. The Chemical Hygiene Plan and MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Graduated cylinders

6.2 Various volumetric flasks (Type A)

6.3 Analytical Balance - OHAUS E-400

6.4 ICP-MS

6.4.1 Perkin Elmer ELAN DRC-e

6.4.2 A radio-frequency generator compliant with Federal Communications Commission (FCC) regulations.

6.4.3 A variable speed peristaltic pump

6.4.4 A mass-flow controller on the nebulizer gas supply is required.

6.4.5 Autosampler - Elemental Scientific, Inc.-SC-4 Fast

6.4.6 Dynamic Reaction Chamber

6.5 A high purity (99.99%) argon gas supply.

6.6 Element DataSystem® LIMS by Promium

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7.0 Reagents and Standards

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

7.1 All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date five years from receipt. All lab prepared regents must be assigned an expiration date one year from preparation.

7.**2** Reagents

Note: Reagents may contain elemental impurities that might affect the integrity of analytical data. Owing to the high sensitivity of Inductively Coupled Plasma - Mass Spectrometry (ICP-MS), high-purity reagents should be used whenever possible. All acids used must be of ultra high-purity grade. Suitable acids are available from a number of manufacturers or may be prepared by sub-boiling distillation. Nitric acid is preferred for ICP-MS in order to minimize polyatomic ion interferences. Several polyatomic ion interferences result when hydrochloric acid (HCl) is used, however, it should be noted that HCl is required to maintain stability in solutions containing antimony and silver. When HCl is used, corrections for the chloride polyatomic ion interferences must be applied to all data.

7.2.1 Reagent Water-All water used during preparation should be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20th Edition of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

7.2.2 Nitric Acid – Concentrated, distilled (specific gravity 1.41).

7.**2**.2.1 Nitric acid (1:1) - Add 50 milliliters (mL) conc. HNO_3 to approximately 30 mL of reagent water and dilute to 100 mL.

7.**2**.3 Hydrochloric acid – Concentrated, distilled (specific gravity 1.19).

7.**2**.3.1 Hydrochloric acid (1:1) - Add 50 mL conc. HCl to approximately 30 mL of reagent water and dilute to 100 mL.

7.3 Stock Standard Solutions - commercially available

Note: Stock standard solutions may be purchased from a reputable commercial source, such as High Purity or NSI, or prepared from ultra high-purity grade chemicals or metals (99.99-99.999% pure).

7.3.1 Refer to the QC/Standards Prep for ICP-MS Logbook, Attachment 2, for details on standard and QC sample sources and preparation.

7.3.2 Label and refer to these solutions as Calibration Standards.

7.3.3 Calibration standards must be verified using an independent source (EPA Quality Control sample) immediately after instrument calibration.

7.4 Working Standards

7.**4**.1 Multi-elemental Calibration Standard Solutions

Care must be taken in the preparation of multi-elemental calibration standard solutions to ensure that the elements are compatible and stable. Fresh calibration standards should be prepared weekly. (Calibration blanks are prepared daily.)

Note: Reference to all working standards can be found in Attachment 2.

7.**4**.1.1 S0/ICB/CCB

Place into a 100 mL volumetric flask 1.0 mL of HNO_3 and 1.0 mL of Internal Standard Solution (See Section 7.3.2). Fill to volume with reagent water.

The resulting concentration of the analytes in the ICB/CCB must be less the PQL. For DoD QSM, the concentrations of the analytes must be ≤ LOD.

7.**4**.1.2 6020-MS1/LLICV/LLCCV

Place into a 200 mL volumetric flask, 2.0 mL of HNO_3 and 2.0 mL of Internal Standard. Pipet 0.200 mL of CP-MS-CRQL into the flask. Fill to volume with reagent water.

7.**4**.1.3 6020-MS2

Place into a 100 mL volumetric flask, 1.0 mL of HNO_3 and 1.0 mL of Internal Standard. Pipet 0.200 mL of CP-MS-CRQL into the flask. Fill to volume with reagent water.

7.**4**.1.4 6020-MS3

Place into a 100 mL volumetric flask, 1.0 mL of HNO_3 and 1.0 mL of Internal Standard. Pipet 0.125 mL of CAL Standard into the flask. Fill to volume with reagent water.

7.**4**.1.5 6020-MS4

Place into a 100 mL volumetric flask, 1.0 mL HNO_3 and 1.0 mL of Internal Standard. Pipet 0.250 mL of CAL Standard into the flask. Fill to volume with reagent water.

7.**4**.1.6 6020-MS5

Place into a 100 mL volumetric flask, 1.0 mL HNO_3 and 1.0 mL of Internal Standard. Pipet 0.500 mL of CAL Standard into the flask. Fill to volume with reagent water.

7.**4**.1.7 CCV

Place into a 200 mL volumetric flask, 2.0 mL HNO_3 and 2.0 mL of Internal Standard. Pipet 0.600 mL of CAL Standard into the flask. Fill to volume with reagent water.

7.**4**.2 Internal Standard Solution

Place in a 100 mL volumetric flask, 50 mL of deionized water and 1.0 mL of HNO_3. Pipet 2.0 mL of Lithium6 single element standard, 1.0 mL of Scandium single element standard, 1.0 mL of Indium single element

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standard, 1.0 mL of Terbium single element standard, and 1.0 mL of Bismuth single element standard. Dilute to volume with reagent water.

Resultant Concentrations: Lithium 20,000 ppb, Scandium 10,000 ppb, Indium 10,000 ppb, Terbium 10,000 ppb, and Bismuth 10,000 ppb

7.4.3 Tuning Solutions

These solutions are used for instrument optimization, tuning, stability, and resolution prior to analysis.

7.**4**.3.1 Smart Tune Solution

This solution is used during the optimization of the ICP-MS (autolen tuning, gas flow, detector voltages, torch box alignment, mass calibration, RF power, and pole bias)

Smart Tune Solution is obtained from Perkin Elmer as a working solution.

Resultant Concentration: 10 ppb of Ba, Be, Ce, Co, In, Mg, Pb, Rh, and U.

7.**4**.3.2 Pre-Calibration Tune Solution

Place into a 100 mL volumetric flask, 1.0 mL of Pre-Tuning Solution, and 1.0 mL of distilled nitric acid. Dilute to volume with reagent water. This solution is used to check the resolution and stability of the ICP-MS.

Resultant Concentration: 100 ppb of Be, Mg, Co, In, and Pb

7.4.4 Initial Calibration Verification (ICV)

The ICV solution independently verifies the accuracy of the initial calibration for each target analyte. The ICV is **purchased** from **Inorganic Ventures** and is a second source standard. Refer to Attachment 2 for all resultant analyte concentrations.

Place into a 200 mL volumetric flask, 2.0 mL distilled nitric acid and 2.0 mL Internal Standard. Pipet **0.4** mL **each** of IC**CV-6 and ICCV-4** multi-element solution.

7.**4**.5 Interference Check Sample (ICS)

This solution is for verifying that correction equations for elemental or polyatomic isobaric interferences are being properly applied. The coefficients in the correction equations were calculated using natural isotopic abundances, and assuming zero instrumental fractionation. However; the correction equation shall not be applied if the appropriate interference check sample measurement demonstrates absence of interferences above the PQL. Refer to Attachment 2 for resultant analyte concentrations.

Interference Check Sample consists of two solutions: ICSA containing only the interferents; ICSAB contains both the interferents and target analytes.

7.**4**.5.1 ICSA Solution

Place into a 200 mL volumetric flask, 2 mL distilled nitric acid and 2 mL Internal Standard. Pipet 20 mL of ICS Part A into flask. Dilute to volume with reagent water.

7.**4**.5.2 ICSAB Solution

Place in a 200 mL volumetric flask, 2 mL distilled nitric acid and 2 mL Internal Standard. Pipet 20 mL of ICS Part A and 20 mL of ICS Part B into flask. Dilute to volume with reagent water.

7.4.6 Low Level Initial Calibration Verification/Low Level Continuing Calibration Verification (LLICV/LLCCV) Standard

The concentrations of the analytes in the LLICV/LLCCV Standard are at the RL.

Place into a 200 mL volumetric flask, 2.0 mL distilled nitric acid and 2.0 mL Internal Standard. Pipet 0.2 mL of CP-MS-CRQL into the flask. Dilute to volume with reagent water.

7.4.7 Method Detection Limit Solution (MDLs)

The MDL Solution shall be a concentration of 3 to 5 times the expected MDL values.

Refer to Standards/QC Preparation LDR/MDL/IDL for ICP-MS for preparation procedures and actual analytes concentrations. (Attachment 4)

7.4.8 Instrument Detection Limits Solution (IDLs)

The IDL Solution shall be prepared at a concentration of 3 to 5 times the instrument manufacturer's suggested instrument detection limits.

Refer to Standards/QC Preparation LDR/MDL/IDL for ICP-MS for preparation procedures and actual analyte concentrations. (Attachment 4)

7.4.9 Linear Dynamic Range (LDR)

The LDR solution determines the linearity of the instrument for each analyte. Due to the varying response signals of the analytes the LDR varies for each analyte; several solutions are prepared in order to meet the criteria.

Refer to Standards/QC Preparation LDR/MDL/IDL for ICP-MS for preparation procedures and actual analytes concentrations. (Attachment 4)

8.0 Sample Preservation and Storage

8.1 All samples must be collected in glass or polyethylene containers. Water/aqueous samples must be preserved with nitric acid to pH less than 2 immediately after collection.

8.2 For the determination of dissolved metals, the sample must be filtered through a 0.45 micrometer (μm) pore diameter membrane filter at the time of collection or as soon as possible. Use a portion of the sample to rinse the filter flask, discard this portion, and collect the required volume of filtrate. Preserve the filtrate with nitric acid to pH less than 2 immediately after filtration.

8.3 Acid preservation of the sample must occur at least 24 hours prior to sample analysis.

8.4 After 60 days the samples may be disposed of in a manner that complies with all applicable regulations.

8.5 Sample digestates must be stored until 365 days after delivery of the data report.

8.6 The maximum holding time for metals is 180 days from sampling.

9.0 Quality Control

9.1 Linear Dynamic Range (LDR)

9.1.1 For all ICP-MS analyses, a LDR check standard must be analyzed and reported. The upper limit of the linear calibration range is established for each analyte by determining the signal responses from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range used for the analysis of samples is determined from the resulting data. The upper LDR limit is an observed signal no more than 10% below the level extrapolated from lower standards.

9.1.2 Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and re-analyzed. The LDRs must be verified whenever a change in instrument hardware operating conditions indicate they should be re-determined, or verified quarterly.

9.13 To meet the requirements of the **DoD**-QSM, the linear dynamic range of the instrument must be verified every 6 months by analyzing a high level standard. The recovery of the analytes must be within ± 10% of the true value.

9.2 Initial Calibration Verification (ICV)

9.2.1 Analyze the ICV standard following the initial calibration, before any samples are analyzed.

9.2.2 The results of the ICV must agree within 10% of the true values for each analyte.

9.2.3 The ICV solution is run at each mass used for reporting final results.

9.2.4 Follow the ICV with the Initial Calibration Blank (ICB).

9.2.4.1 Any analyte present in the blank must be less than the PQL.

9.2.4.2 To meet the requirements of the **DoD**-QSM, any analytes detected in the ICB must be ≤ LOD.

9.2.5 If criteria are not met, the run must be terminated and the instrument re-calibrated. If re-calibration does not fix the problem, shut down the instrument and request instrument service.

9.3 Low Level Initial Calibration Verification/Low Level Continuing Calibration Verification (LLICV/LLCCV) Standard

9.3.1 To verify linearity near the RL, a standard at the RL (LLICV) is prepared, in the same acid matrix as the calibration standards, and analyzed at the beginning of each sample analysis run.

9.3.2 The LLICV is run for every required isotope used for the analysis of all Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) analytes. Information regarding the LLICV is reported on Form II.

9.3.3 If the percent recovery of the LLICV falls outside the control limits of 70 - 130% for one or more analytes, the LLICV is re-analyzed immediately (after corrective action) for those analytes only. If the results of the re-analysis for those analytes fall within the control limits, no further corrective action is required. If the results of the re-analysis for those analytes do not fall within the control limits, the analysis is terminated, the problem corrected, the instrument recalibrated, the LLICV analyzed, and the samples associated with the LLICV re-analyzed. The above criteria also apply to the LLCCV standard.

9.3.4 A LLCCV standard is run after every 19 samples and at the end of the sequence.

9.4 Interference Check Sample

9.4.1 Follow the ICB analysis with the Interference Check samples (ICSA and ICSAB, consecutively). The Interference Check samples are analyzed at the beginning of the analytical run. The Interference Check samples should be followed, immediately, by the analysis of a LLCCV and a CCV/CCB pair. The ICSA and ICSAB are also run every 12 hours.

9.4.2 The Interference Check samples consist of two solutions: Solution A (ICSA) and Solution AB (ICSAB). Solution A contains the interferents and Solution AB consists of the analytes and interferents (see Attachment 5).

9.4.3 The analytical results of ICS Solution A (ICSA) shall fall within the control limit of ± 3 times the PQL of the analyte's true value or ± 20% of the analyte's true value (the true value is zero unless otherwise stated) in the ICSA, whichever is greater. If not, the analysis is terminated, the problem corrected, the instrument recalibrated, and the analytical samples re-analyzed. The ICSA results for these analytes are reported from an undiluted sample analysis

9.4.4 ICS reference material may be obtained from EPA or an independent source such as ERA. Because the values may vary with each lot purchased, the analyte concentrations shown in the table below are typical and provided as an example only. Certificates of analysis with true values for each lot are maintained in a three ring binder in the laboratory.

9.4.5 Results for the ICS Solution AB (ICSAB) during the analytical runs shall fall within the control limit of ± 3 times the PQL of the true value or ± 20% of the true value, whichever is greater, for the analytes included in the ICSAB. If not, the analysis is terminated, the problem corrected, the instrument recalibrated, and the analytical samples re-analyzed.

9.4.6 To meet the requirements of the **DoD**-QSM, the absolute value of all analytes not spiked in the ICS-A must be < LOD, unless the analyte is a verified trace impurity from one of the spiked analytes. The analyte recoveries in the ICS-AB must be within ± 20%.

9.4.6.1 If the ICS do not meet the criteria in Section 9.4.6, terminate analyses. Correct problem and reanalyze ICS and any associated samples.

9.4.6.2 If corrective actions fail, qualify data in the narrative. Refer to the **DoD**-QSM "Q" flag.

9.5 Continuing Calibration Verification (CCV)

9.5.1 Verify the calibration at the beginning of the analytical sequence, every 10 samples, and at the end of analytical run.

9.5.2 The results of the check standard must agree within ±10% of the true value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

9.6 Continuing Calibration Blank (CCB)

9.6.1 A calibration blank must be analyzed immediately after each initial and continuing calibration verification standard at a frequency of 10%.

9.6.2 Calibration blanks are prepared using same acid volumes as calibration standards.

9.6.3 Any analyte present in the CCB must be less than the RL.

9.6.3.1 To meet the requirements of the **DoD**-QSM, analytes in the CCB must be ≤ LOD.

9.6.3.2 If the CCB fails the criteria in Section 9.6.3.1, re-prepare and reanalyze the CCB and the previous 10 samples.

9.6.4 If criteria are not met, terminate the analysis and recalibrate. If re-calibration does not fix the problem, shut down the instrument and request instrument service.

9.7 Preparation Blank Analysis

9.7.1 A preparation blank is prepared with each digestion batch of up to 20 samples.

9.7.2 This blank is used to ascertain whether sample concentrations reflect contamination.

9.7.3 The blank must contain absolute values of analyte concentrations at less than or equal to the RL.

9.7.3.1 To meet the requirements of the **DoD**-QSM, the concentrations of any analytes in the preparation blank must be ≤ ½ the RL.

9.7.3.2 If the preparation blank fails the criteria in Section 9.7.3.1, re-prepare and analyze the associated samples.

9.7.4 If the blank exceeds the RL, the entire sample batch associated with the blank must be re-prepared along with a new preparation blank. The only circumstance under which the method blank can be reported with analyte values greater than the RL is when the concentration of the affected analyte in the sample exceeds the blank concentration by a factor of 10 or more or all samples are less than the PQL.

9.8 Spiked Sample

9.8.1 A matrix spike and matrix spike duplicate must be prepared with each SDG.

9.8.2 If the sample concentration is less than four times the amount spiked and the recovery of the spike is less than 75% or greater than 125%, the corresponding element is flagged with an "N" on Form 1 to indicate that the element did not recover in the matrix spike acceptably (See Attachment 3 for spiking levels.)

9.8.3 Prepare and analyze a Post Digestion Spike (PDS) for those elements flagged with an "N".

The PDS should be spiked between 10 – 100X the RL.

9.8.4 To meet the requirements of the **DoD**-QSM, the matrix spike is evaluated with the same control limits as the LCS (± 20%). The RPD between duplicate matrix spikes or duplicate sample should be < 20%.

9.8.4.1 If the duplicate matrix spikes do not meet the criteria in Section 9.8.4, contact the client for guidance. Qualify the specific analytes in the original sample in the narrative. Refer to the **DoD**-QSM "J" flag.

9.9 Duplicate Sample

9.9.1 A duplicate sample analysis should be performed once per SDG.

9.9.2 If the sample and duplicate do not agree within a 20% RPD when the concentration is either greater than or equal to 5 X RL, or ± RL when the concentration is < 5 X PQL, then the affected element is flagged with an "*" to indicate poor duplication of results.

9.9.3 See Section 9.8.4 for the **DoD**-QSM duplicate sample criteria.

9.10 Laboratory Control Sample (LCS)

9.10.1 A water/aqueous Laboratory Control Sample is prepared with each digestion batch of up to 20 samples.

9.10.2 The results of the aqueous LCS must agree within ± 20% of the true values for all the elements.

If the aqueous LCS falls outside these control limits, the samples associated with that LCS must be re-digested and reanalyzed.

9.11 Serial Dilution

9.11.1 One serial dilution must be performed for each SDG.

9.11.2 Perform a 5x serial dilution on the sample to determine if a chemical or physical interference exists.

9.11.3 If the analyte concentration is 50 times or more above the instrument detection limit in the original sample, the serial dilution must then agree within 10% of the original sample. If not, then flag the appropriate elements with an "E" to indicate that an interference exists. The “E” flag is not used for DOD-QSM analyses.

9.11.4 To meet the requirements of the **DoD**-QSM, a 5-fold dilution is performed for each preparatory batch on a sample with analyte concentration (s) > 50X LOQ. The dilution must agree within ± 10% of the original sample.

9.11.4.1 If the serial dilution fails, a post-digestion spike (PDS) is performed.

9.11.4.2 The recovery for the PDS must be within the 75-125% of the expected value.

9.11.4.3 If the PDS fails, qualify the sample results as estimates in the narrative. Refer to the **DoD**-QSM “J” flag.

9.12 Field Blanks

9.12.1 Samples identified as field blanks should not be used for sample spike, duplicate, or serial dilution analysis.

9.13 Sample Tracking Records

9.13.1 Each sample bottle must be labeled with a CompuChem sample identification number.

9.14 Instrument Printout

9.14.1 Because of the nature of the data being collected, all analytical data generated must be labeled so that it can be identified.

9.14.2 Note any dilutions, duplicate analysis, spike analysis, and blank and standard analyses.

9.15 Internal Standards

9.15.1 The analyst shall monitor all responses from the internal standards through the analytical run. The absolute response of any ONE internal standard must not deviate more than 30 – 120% of the original response in the calibration blank for Method 6020 and **DoD**-QSM analyses. For Method 6020A, corrective action is required if the response of any internal standard falls below 70%. If deviations greater than these are observed in field samples, matrix spikes, or duplicate samples, the original sample shall be diluted by a factor of five, internal standard added, and the sample reanalyzed. Continue to dilute sample by factors of 5, until the Internal Standard comes within control.

9.16 Contingency

9.16.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.16.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.16.3 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 The instrument calibration standards are analyzed at the beginning of the analytical sequence, in the order shown in the ICP-MS Run Log (Attachment 1) and must meet acceptance criteria before samples can be analyzed. Calibration is further discussed in the Procedure section that follows.

10.2 A six-point calibration curve (initial calibration blank and 5 concentration levels) is prepared for the ICP-MS. This calibration is performed daily. To meet the requirements of the **DoD**-QSM and Method 6020, the correlation coefficient of the calibration curve must be ≥ 0.995. For Method 6020A, the correlation coefficient must be ≥ 0.998.

10.3 The calibration curve is verified by the ICV and CCV. The ICV and CCV must meet acceptance criteria or a new calibration is required.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

11.1 Pre-Tune Optimization

Prior to running any analytical sequence, the instrument parameters MUST be optimized.

11.1.1 Alignment of the Sampler and Skimmer Cones.

11.1.1.1 Aspirate the "Smart Tune Solution."

11.1.1.2 Under the "Method" tab, open the workspace "X-Y alignment" and run the method.

11.1.1.2 Select the "Realtime" display and rotate the X-Y alignment controls clockwise and counter-clockwise until a maximum count for In 115 is achieved.

11.1.2 Optimization of the Autolen, Nebulizer Gas Flow, Lens Voltage, and Detector Voltages.

11.1.2.1 Aspirate the "Smart Tune Solution."

11.1.2.2 Under the "Smarttune" workspace, open "Smarttune OPT1" and run the workspace.

Note: The workspace contains the manufacturer's recommended optimization ranges. During the acquisition phase, the instrument will be optimizes for each of these parameters. If the criteria cannot be met, a "failed" message will appear. Refer to the instrument manual or call the Perkin Elmer Help hotline to correct failing parameters.

11.1.3 Dual Detector Calibration.

Dual Detector Calibration is achieved by producing 1 – 3 million counts for each target element. Since some elements are more sensitive than others, the solution concentration varies for each element and from each calibration attempt.

A valid Dual Detector Calibration contains at least 21 data points and a correlation coefficient of ≥ 0.998. The summary page displays the number of data points and the correlation coefficient used to determine the Dual Detector coefficient. If this criterion is not met for all target elements, adjust the analyte concentration in the solution and re-determine the Dual Detector Calibration coefficients.

11.1.3.1 Aspirate the Dual Detector Calibration solution.

11.1.3.2 Under the "Smarttune" workspace, open "SmartTune Dual1" and run the workspace.

11.1.3.3 Verify that all criteria have been met for the target elements. Save and load the workspace.

11.2 Tune Verification

Prior to running any analytical sequence, the mass resolution and stability must be verified.

Tune verification is achieved by analyzing 5 replicates of the 100 ppb Tune Solution. The %RSD for the 5 replicates must be < 5%. The mass resolution must be verified to be within 0.1 amu over the mass range of 6 – 120 amu and the peak width at 10% peak height must be within 0.60 – 0.80 amu.

11.2.1 Mass Resolution

11.2.1.1 Aspirate the "100 PPB Tune Solution."

11.2.1.2 Under the "Smarttune" workspace, open "SmarttuneRES1" and run the workspace.

Note: The workspace contains the manufacturer's recommended AMU ranges. During the acquisition phase, the instrument will adjust the DAC values to meet criteria. Refer to the instrument manual or call the Perkin Elmer Help hotline to correct failing parameters.

11.2.1.3 Save the parameter by opening the "Tuning" window on the menu bar.

11.2.2 Instrument Stability

11.2.2.1 Aspirate the "100 PPB Tune Solution."

11.2.2.2 From the menu bar, select the Worksheet window and open "6020 TUNE" located in the 6020 folder. Open the report tab, and define the file name of the tune as PEMSATUNEMMDDYY and save the worksheet.

11.2.2.3 From the menu bar, select the Sample window. Under the Manual tab, populate the sample field with "TUNE" and populate the detail field with "100 PPB Tuning Soln at 10% Peak Height".

11.2.2.4 Run the workspace by clicking on the "Analyze Sample" button. Once completed verify that the %RSD does not exceed 5 percent. If so, refer to instrument manual or call Perkin Elmer Help hotline for help.

11.3 Instrument Calibration and Sample Analysis

Prior to running the calibration, a method was built to contain all instrument operating and acquisition parameters, configurations, timing parameters, and calibration and verification sequences in accordance with the SW-846 Methods 6020/6020A.

11.3.1 From the menu bar, select the Worksheet window and open "6020AA" located in the 6020 folder. Open the report tab, and define the file name of the sequence file as PEMSIIIMMDDYY and save the worksheet.

11.3.2 From the menu bar, select the Sample window and open the Sample tab.

11.3.3 From the desktop, launch "Sample Table" and enter in the preparation batch number. Hit "Build ICPMS Table". Using this sample table populate the ELAN's sample table fields with the following: Batch ID field with SDG number, the Sample ID field with the sample IDs, and the Description field with the client ID. In the "Measure Action" column select the command "Run Blank, Stds, and Sample." Save the Autosampler table as the same name as the sequence file name and print. Label and pour up sample tubes according to Autosampler table

11.3.4 Begin the Analyses by selecting the samples of interest and clicking on Analyze Batch.

11.3.5 Once analyses are complete, the data undergo a peer review process to verify all acceptance criteria have been met. Samples associated with failing acceptance criteria are re-analyzed.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.2 Calculation of the standard deviation of a set of values:

$$\text{Standard deviation} = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.3 Calculation of percent recovery:

12.3.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\,100$$

12.3.2 Matrix spikes:

$$\%\,\mathrm{R} = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\,100$$

12.4 Calculation of % RSD

$$\%\mathrm{RSD} = \left(\frac{Standard\ deviation}{\overline{\mathrm{X}}}\right) \times 100$$

12.5 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{\left(Value\ 1\ +\ Value\ 2\right)/2}\ x100$$

12.6 Calculation of %Difference (%D)

$$\%Diff = \frac{Value\ - \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.7 Concentration of aqueous samples

$$\mu g / L = \frac{(C)(V)(D)}{(W)}$$

where: C - Concentration (mg/l) D- Dilution factor
V - Final volume of digestate (L) W - Volume of sample (L)

12.8 Calculating Dilutions

If a sample concentration exceeds the linear range of the instrument, a dilution must be performed. A 10x dilution is performed using 1 mL sample plus 9 mL diluent for a total volume of 10 mL. It should be recorded on the run log as "10x (1 mL in 10 mL)."

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for a single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96 and Update IV 2/07, Methods 6020/6020A

16.2 ICP-MS Operating Manual for VG PQ Excell Thermo Elemental

16.3 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.4 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.5 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 **TNI** Standards, effective July **2011**, plus revisions

16.7 EPA QA/G6: Guidance for Preparing Standard Operating Procedures EPA/600/B-07/001, April 2007.

16.8 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **October 25, 2010**

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 Attachment 1 – Example ICP-MS Run Log

17.2 Attachment 2 – Example Standards/QC Preparation for ICP-MS

17.3 Attachment 3 – Spiking Levels

17.4 Attachment 4 – LDR/MDL/IDL Preparation Sheets

17.5 Attachment 5 – Certified Values for Interference Check Sample for ICP-MS

Attachment 1

Daily Instrument
Nebulizer pressure: _____
Peak position: _____
Peak intensity: _____
Vernier position: _____

Maintenance: _____

Printed:

Method(s (circle **ILM05.4** **6010B** **6010C** **Other** _____

Operator(s) _____ **Date/Time** _____ **to** _____

↑	Specified element(s) fail(s) high
↓	Specified element(s) fail(s) low
RR	Re-run sample(s) neat
RR #X	Re-run at a #-fold dilution
#X	Sample run at a #-fold dilution (aliquot of sample diluted with acidified water)
L	Matrix = leachate
W	Matrix = water
S	Matrix = solid
B	Matrix = biological tissue

SDG	Matrix	Comments
1001011		
1001015		

ICP Run Log

File Name:

Sample ID	Client ID	Sample	DF	Date	Time	SDG	Action Needed

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Reviewed By: _____ Date: _____ Page 1 of 1

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If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 2

Standards/QC Preparation for ICP-MS (PEMS) 1 of 3 CompuChem a division of Liberty Analytical Corp. **WORKSHEET 1 AA(1)**

STANDARDS and REAGENTS TRACEABILITY Seq. File Name: _____.

STANDARD / REAGENT	Reference # *	Manufacturer	Lot #	Expiration Date
Nitric Acid (HNO3)	NHO3-D-112009-DDD	JT Baker	H12022	11/10
Hydrochloric Acid (HCl)	HCL-D-101209-DDD	Caledon	71129	10/10
ICP-MS-TS-3 (Pre Tune Soln)	9I04001	High Purity	0904345	08/28/10
Q-6042-M (CAL Standard)	0B18009	NSI	020310	02/28/12
ICV-1 (0307) multi-element solution	9L15002	EPA	0307	12/10
SM-770-032 (CP-MS-CRQL-Rev multi-element solution)	9J22003	High Purity	0928516	10/12/10
ICSA (0803) multi-element solution	9C30002	EPA	0803	12/10
ICSAB (0803) multi-element solution	9C30003	EPA	0803	12/10
LITHIUM^6 (Li^6) 1000 mg/L single element standard	0B16007	High Purity	0911701	07/20/11
SCANDIUM (Sc) 1,000 mg/L single element standard	8L17019	High Purity	0801101	06/15/10
INDIUM (In) 1,000 mg/L single element standard	8L17017	High Purity	0808613	06/15/10
TERBIUM (Tb) 1,000 mg/L single element standard	8L17018	High Purity	0811919	06/15/10
BISMUTH (Bi) 1,000 mg/L single element standard	0B16008	High Purity	1000418	07/20/11
TIN (Sn) 10 µg/mL single element standard	7M1-42-12	NSI	1020008-SN	10/10
MOLYBDENUM (Mo) 10 µg/mL single element standard	7M1-42-13	NSI	1020008-MO	10/10
Water Spike Sample D ICP-MS Matrix Spike multi-element solution	9I15001	High Purity	0934201	12/10/10

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log

Multi-Element Stock Standard Concentrations:

Stock Standard	Analyte Concentrations (µg/mL)
Pre Tune Solution	Be: 10 Mg: 10 Co: 10 In: 10 Pb: 10
CAL standard	Al: 1000 Sb: 100 As: 100 Ba: 100 Be: 100 B: 100 Cd: 100 Ca: 1000 Cr: 100 Co: 100 Cu: 100 Fe: 1000 Pb: 100 Mg: 1000 Mn: 100 Mo: 100 Ni: 100 K: 1000 Se: 100 Si: 100 Ag: 100 Na: 1000 Tl: 100 Ti: 100 V: 100 Zn: 100 Sn: 100 Sr: 100
CP-MS-CRQL-Rev multi-element solution	Be: 1 V: 5 Cr: 2 Mn: 1 Co: 1 Ni: 1 Cu: 2 Zn: 2 As: 1 Se: 5 Ag: 1 Cd: 1 Sb: 2 Ba: 10 Tl: 1 Pb: 1 Al: 30 Sn: 1 Mo: 1

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Reviewed By: _____ Date: _____

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Attachment 2 (continued)

Standards/QC Preparation for ICP-MS (PEMS) 2 of 3 CompuChem a division of Liberty Analytical Corp. **WORKSHEET 1 AA(1)**

Standard Lot Number *	Preparation Instructions	Resultant Analyte Conc. (µg/L)
Instrument Tune Solution– 02 16 09- JCF	Place in a 500-mLvolumetric flask: 0.5 mL of PRE-Tuning solution and 5 mL distilled nitric acid. Dilute to volume with deionized water	Li^7: 10 Be: 10 Mg: 10 Co: 10 In: 10 Ce: 10 Pb: 10 U: 10
Internal Standard– 02 16 10 – JCF	Place in a 100-mLvolumetric flask: 50 mL of deionized water , 1.0 mL of distilled nitric acid. Pipet: 2.0 mL of Lithium^6, 1.0 mL of Scandium, 1.0 mL of Indium, 1.0 mL of Terbium, and 1.0 mL of Bismuth. Dilute to volume with deionized water	Li^6: 20000 Sc: 10000 In: 10000 Tb: 10000 Bi: 10000
S0/ICB/CCB– 03 08 10 – JCF	Place in a 100-mL volumetric flask: 1 mL distilled nitric acid and 1mL Internal Standard. Dilute to volume with deionized water. Prepare Daily.	Li^6: 200 Sc: 100 In: 100 Tb: 100 Bi: 100
S 6020-MS1/LLICV/LLCCV– 03 08 10 – JCF	Place in a 200-mL volumetric flask: 2 mL distilled nitric acid and 2 mL Internal Standard. Pipet 0.200 mL of CP-MS-CRQL, into flask. Dilute to volume with deionized water	Li^6: 200 Be: 1 Al: 30 Sc: 100 V: 5 Cr: 2 Mn: 1 Co: 1 Ni: 1 Cu: 2 Zn: 2 As: 1 Se: 5 Mo: 1 Ag: 1 Cd: 1 In: 100 Sn: 1 Sb: 2 Ba: 10 Tb: 100 Tl: 1 Pb: 1 Bi: 100
S 6020-MS2 –03 08 10 – JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid and 1 mL Internal Standard. Pipet 0.200 mL of CP-MS-CRQL, into flask. Dilute to volume with deionized water.	Li^6: 200 Be: 2 Al: 60 Sc: 100 V: 10 Cr: 4 Mn: 2 Co: 2 Ni: 2 Cu: 4 Zn: 4 As: 2 Se: 10 Mo: 2 Ag: 2 Cd: 2 In: 100 Sn: 2 Sb: 4 Ba: 20 Tb: 100 Tl: 2 Pb: 2 Bi: 100
S 6020-MS3–03 08 10 – JCF	Place in a 100-mL volumetric flask: 1 mL distilled nitric acid and 1 mL Internal Standard. Pipet 0.125 mL of CAL Standard into flask. Dilute to volume with deionized water	Li^6: 200 Be: 125 Al: 1250 Sc: 100 V: 125 Cr: 125 Mn: 125 Co: 125 Ni: 125 Cu: 125 Zn: 125 As: 125 Se: 125 Mo: 125 Ag: 125 Cd: 125 In: 100 Sn: 125 Sb: 125 Ba: 125 Tb: 100 Tl: 125 Pb: 125 Bi: 100 Al: 1250 Ca: 1250 Na: 1250 Mg: 1250 Fe: 1250 K: 1250
S 6020-MS4–03 08 10 – JCF	Place in a 100-mL volumetric flask: 1 mL distilled nitric acid and 1 mL Internal Standard. Pipet 0.250 mL of CAL Standard into flask. Dilute to volume with deionized water	Li^6: 200 Be: 250 Al: 2500 Sc: 100 V: 250 Cr: 250 Mn: 250 Co: 250 Ni: 250 Cu: 250 Zn: 250 As: 250 Se: 250 Mo: 250 Ag: 250 Cd: 250 In: 100 Sn: 250 Sb: 250 Ba: 250 Tb: 100 Tl: 250 Pb: 250 Bi: 100 Al: 2500 Ca: 2500 Na: 2500 Mg: 2500 Fe: 2500 K: 2500

*Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

Standard Solutions Prepared By: _____

Entered By: _____ _____ Date: _____

Reviewed By: _____ Date: _____

032910:jad

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Attachment 2 (continued)

Standards/QC Preparation for ICP-MS (PEMS) 3 of 3 CompuChem a division of Liberty Analytical Corp. **WORKSHEET 1 AA(1)**

Standard Lot Number *	Preparation Instructions	Resultant Analyte Conc. (µg/L)
S 6020-MS5–03 08 10 – JCF	Place in a 100-mL volumetric flask: 1 mL distilled nitric acid and 1 mL Internal Standard. Pipet 0.50 mL of CAL Standard into flask. Dilute to volume with deionized water	Li^6: 200 Be: 500 Al: 5000 Sc: 100 V: 500 Cr: 500 Mn: 500 Co: 500 Ni: 500 Cu: 500 Zn: 500 As: 500 Se: 500 Mo: 500 Ag: 500 Cd: 500 In: 100 Sn: 500 Sb: 500 Ba: 500 Tb: 100 Tl: 500 Pb: 500 Bi: 100 Al: 5000 Ca: 5000 Na: 5000 Mg: 5000 Fe: 5000 K: 5000
ICV– 03 08 10 – JCF	Place in a 200-mL volumetric flask: 2 mL distilled nitric acid, and 2 mL Internal Standard. Pipet 4.0 ml of ICV (0307) multi-element solution, 2.2 mL of 10 µg/mL Molybdenum and 2.2 mL of 10 µg/mL Tin into flask. Dilute to volume with deionized water. (Note: Tin must be from a source different than that used for the calibration standards)	Al: 504 Sb: 199 As: 200 Ba: 99 Be: 99 Ca: 2005 Cr: 98 Co: 100 Cu: 98 Fe: 1016 Pb: 200 Mg: 1215 Mn: 100 Ni: 101 K: 2004 Se: 206 Ag: 100 Na: 2019 Tl: 206 V: 100 Zn: 205 Cd: 99 Li^6: 200 Sc: 100 In: 100 Tb: 100 Bi: 100 Mo: 110 Sn: 110
ICSA– 03 08 10 – JCF	Place in a 200-mL volumetric flask: 2 mL distilled nitric acid and 2 mL Internal Standard. Pipet 20.0 mL of ICS Part A (0803) into flask. Dilute to volume with deionized water.	Al: 100000 Sb: 1.5 As 0.1 Ba: 1.2 Be: 0 Cd: 0.7 Ca: 100000 C: 200000 Cl: 1000000 Cr: 21 Co: 1 Cu: 8 Fe: 10000 Pb: 4 Mg: 100000 Mn: 7 Mo: 2000 Ni: 6 P: 100000 K: 10000 Se: 0.3 Ag: 0 Na: 100000 S: 100000 Tl: 0 Ti: 2000 V: 0.5 Zn: 11, Li^6: 200 Sc: 100 In: 100 Tb: 100 Bi: 100
ICSAB– 03 08 10 – JCF	Place in a 200-mL volumetric flask: 2 mL distilled nitric acid and 2 mL Internal Standard. Pipet 20.0 mL of ICS Part A (0803) and 20.0 mL of ICS Part B (0803) and 0.400 mL of 10 µg/mL Tin into flask. Dilute to volume with deionized water.	Al: 100000 Sb: 22 As 19 Ba: 22 Be: 19 Cd: 20 Ca: 100000 C: 200000 Cl: 1000000 Cr: 40 Co: 20 Cu: 25 Fe: 10000 Pb: 25 Mg: 100000 Mn: 27 Mo: 2000 Ni: 24 P: 100000 K: 10000 Se: 19 Ag: 18 Na: 100000 S: 100000 Tl: 21 Ti: 2000 V: 19 Zn: 29, Li^6: 200 Sc: 100 In: 100 Tb: 100 Bi: 100
CCV– 03 08 10 – JCF	Place in a 200-mL volumetric flask: 2 mL distilled nitric acid, and 2 mL Internal Standard. Pipet 0.600 mL of CAL Standard. Dilute to volume with deionized water.	Li^6: 200 Be: 300 Sc: 100 V: 300 Cr: 300 Mn: 300 Co: 300 Ni: 300 Cu: 300 Zn: 300 As: 300 Se: 300 Mo: 300 Ag: 300 Cd: 300 In: 100 Sn: 300 Sb: 300 Ba: 300 Tb: 100 Tl: 300 Pb: 300 Bi: 100 Al: 3000 Na: 3000 Ca: 3000 K:3000 Mg: 3000 Fe: 3000

*Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

Standard Solutions Prepared By: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

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Attachment 3

Spiking Levels for Spike Sample Analysis

Analyte	Spike (µg/L)
Sb	100
As	40
Ba	2000
Be	50
Cd	50
Cr	200
Co	500
Cu	250
Pb	20
Mn	500
Ni	500
Se	10
Ag	50
Tl	50
V	500
Zn	500

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Attachment 4

Standards Preparation Log for ICP-MS (PEMS) Linear Range Standard (LDR) Studies 1 of 2
Worksheet 1 P(B) Rev. 3

STANDARDS and REAGENTS TRACEABILITY

	Ref #*	Manufacturer	Lot #	Expiration Date
Q-6042-M (CAL standard)	0B18009	NSI	020310	02/01/12
Barium (1000 ppm stock standard)	8L17007	High Purity	0830327	06/15/10
Manganese (1000 ppm stock standard)	8L17012	High Purity	0805617	06/15/10
Zinc (1000 ppm stock standard)	9L31017	High Purity	0916203	06/11/11

Multi-Element Stock Standard Concentrations:

Stock Standard	**Analyte Concentrations (µg/mL)**
CAL standard	Al: 1000 Sb: 100 As: 100 Ba: 100 Be: 100 B: 100 Cd: 100 Ca: 1000 Cr: 100 Co: 100 Cu: 100 Fe: 1000 Pb: 100 Mg: 1000 Mn: 100 Mo: 100 Ni: 100 K: 1000 Se: 100 Si: 100 Ag: 100 Na: 1000 Tl: 100 Ti: 100 V: 100 Zn: 100 Sn: 100 Sr: 100

Entered By: J. FELDHAUS

Reviewed By:_____ Date:_____

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Attachment 4 (continued)

Standards Preparation Log for ICP-MS (PEMS) Linear Range Standard (LDR) Studies 2 of 2
Worksheet 1 P(B) Rev. 3

Standard Lot Num.**	Preparation instructions	True Values (ppb)
LDR-0.5-041310-JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid, and 1 mL Internal Standard. 0.5 ml of CAL Standard into flask. Dilute to volume with deionized water	Sb: 500 As: 500 Ba: 500 Be: 500 Cd: 500 Cr: 500 Co: 500 Cu: 500 Pb: 500 Mg: 500 Mn: 500 Ni: 500 Se: 500 Ag: 500 Tl: 500 V: 500 Zn: 500
LDR-1.0-041310-JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid, and 1 mL Internal Standard. 1.0 ml of CAL Standard into flask. Dilute to volume with deionized water	Sb: 1000 As: 1000 Ba: 1000 Be: 1000 Cd: 1000 Cr: 1000 Co: 1000 Cu: 1000 Pb: 1000 Mg: 1000 Mn: 1000 Ni: 1000 Se: 1000 Ag: 1000 Tl: 1000 V: 1000 Zn: 1000
LDR-2.0-041310-JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid, and 1 mL Internal Standard. 2.0 ml of CAL Standard into flask. Dilute to volume with deionized water	Sb: 2000 As: 2000 Ba: 2000 Be: 2000 Cd: 2000 Cr: 2000 Co: 2000 Cu: 2000 Pb: 2000 Mg: 2000 Mn: 2000 Ni: 2000 Se: 2000 Ag: 2000 Tl: 2000 V: 2000 Zn: 2000
LDR-3.0-041310-JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid, and 1 mL Internal Standard. 3.0 ml of CAL Standard into flask. Dilute to volume with deionized water	Sb: 3000 As: 3000 Ba: 3000 Be: 3000 Cd: 3000 Cr: 3000 Co: 3000 Cu: 3000 Pb: 3000 Mg: 3000 Mn: 3000 Ni: 3000 Se: 3000 Ag: 3000 Tl: 3000 V: 3000 Zn: 3000
LDR-4.0-041310-JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid, and 1 mL Internal Standard. 4.0 ml of CAL Standard into flask. Dilute to volume with deionized water	Sb: 4000 As: 4000 Ba: 4000 Be: 4000 Cd: 4000 Cr: 4000 Co: 4000 Cu: 4000 Pb: 4000 Mg: 4000 Mn: 4000 Ni: 4000 Se: 4000 Ag: 4000 Tl: 4000 V: 4000 Zn: 4000
LDR-MnBaZn-041310-JCF	Place in a 100-mL volumetric flask: Pipet 1.0 mL distilled nitric acid, and 1 mL Internal Standard. 1.0 ml of Ba, Mn, and Zn 1000 ppm stock standard into flask. Dilute to volume with deionized water	Ba: 10000 Mn: 10000 Zn: 10000

**Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

Applies to Seq. File(s): PEMSJCF041310

Standard Solutions Prepared By: _____

Entered By: J. FELDHAUS

Reviewed By: _____ Date: _____

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Attachment 5

"Certified Values" for Interference Check Sample ICP-MS
Part A (0803) and Part A (0803) Mixed with Part B (0803)

Element	**Part A** µg/L	**Part A + B** µg/L
Al	[100000]	[100000]
SB	(1.5)	22
Ba	(1.2)	22
Be	(0)	19
Cd	(0.7)	20
Ca	[100000]	[100000]
C	[200000]	[200000]
Cl	[1000000]	[1000000]
Cr	21	40
Co	1	20
Cu	8	25
Fe	[100000]	[100000]
Pb	4	25
Mg	[100000]	[100000]
Mn	7	27
Mo	[2000]	[2000]
Ni	6	24
P	[100000]	[100000]
K	[100000]	[100000]
Se	(0.3)	19
Ag	(0)	18
Na	[100000]	[100000]
S	[100000]	[100000]
Tl	(0)	21
Ti	[2000]	[2000]
V	(0.5)	19
Zn	11	29

[] Indicates analytes that do not require ICP-MS Determination in the ICS.

() Indicates analyte values that are less than the CRQL and the value is to be used as a set point for +/- 3 times CRQL acceptance criteria calculations.

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SOP 3.3.4

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: _____ SOP Section #: 3.3.4 Revision #: 24

SOP Title: Automated Cold Vapor Determination for Mercury by CLP, SW-846 and MCAWW

Effective date: (QA fills in) 4/6/11

- Procedure prepared by: Judith Mozer Date: 4/6/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [illegible] Date: 04/06/11
- Reason for change: annual review
- This procedure meets the requirements of the following approved method references: USEPA CLP SOW ILM05.4, SW-846 3RD Edition, Updates III and IV, 9/94 and 2/07, Method 7470A, Methods 7471A/7471B, MCAWW, 1994, Method 245.1, Rev. 3.0, DoD-QSM, Version 4.2, October 25, 2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 005: Automated Cold Vapor Determination for Mercury by CLP, SW-846, and MCAWW

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Instrument Procedure 005: Automated Cold Vapor Determination for Mercury by CLP, SW-846, and MCAWW

1.0 Scope and Application

1.1 This Standard Operating Procedure (SOP) describes the determination mercury in drinking and surface waters, as well as soil and sediment samples after appropriate preparation. It may be applicable also to saline waters, waste waters, effluents, and domestic sewages, providing potential interferences are not present.

1.2 The working range is 0.20 to 10.0 µg Hg/L

1.3 Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

1.4 This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

2.1 The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 ηm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.2 In addition to inorganic forms of mercury, organic mercurials may also be present. These organo-mercury compounds will not respond to the flameless atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenyl mercuric acetate and methyl mercuric chloride, are only partially oxidized by the reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant for these compounds.

Therefore, a persulfate oxidation step following the addition of the permanganate was included during digestion to ensure that organo-mercury compound(s), if present, will be oxidized to the mercuric ion before measurement.

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3.0 Definitions

3.1 Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte **(40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL $<$ LOD $\leq$ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported between the DL and the LOQ must be flagged as estimated values.

3.6 Reporting Units – µg/L for water and mg/Kg for soil

3.7 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.8 SC DHEC – South Carolina Department of Health and Environmental Control

3.9 CRQL – Contract Required Quantitation Limit (for ILM05.4)

3.10 **DoD**-QSM – Department of Defense Quality Systems Manual

3.11 Digestion Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.12 LIMS – Laboratory Information Management System

4.0 Interferences

4.1 Some sea waters, waste waters, and sediment samples which are high in chlorides have shown a positive interference, probably due to the formation of free chlorine which will absorb radiation at 253 ηm.

4.2 Interference from certain volatile organic materials that will absorb at this wavelength is also possible. A preliminary run under oxidizing conditions, without stannous chloride, would determine if this type of interference is present.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation, glasses, gloves and lab coats are a minimum requirement. The

persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory. The Chemical Hygiene Play and the MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 Atomic Absorption Spectrophotometer: Leeman Labs, Inc. HYDRA AA/PS200II Automated Mercury Analysis System **with Win Hg Runner Software, Version 1.7**

6.2 Volumetric flasks (100-mL, 1-L, and 2-L)

6.3 13 x 100 mm glass culture tubes

6.4 Element DataSystem® LIMS computer software by Promium

7.0 Reagents and Standards

Record all reagent and standard preparations in the Standards/QC Preparation Log for Cold Vapor Mercury (Attachment 1) **or in LIMS**. All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date **five** years from receipt date. All lab prepared regents must be assigned an expiration date one year from preparation date.

Note: All spiking standard information is entered into the LIMS. To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent. All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent water- All water used during preparation should be reagent-grade Type I with regard to resistivity of >10 megohm-cm (20th Edition of standards Methods, Method 1080), and referred throughout this SOP as reagent water.

7.2 Stannous chloride (10%) - Dissolve 100 grams into 500 mL of reagent water contained in a 1 L volumetric flask. Add 250 mL of concentrated HCl and dilute to volume with reagent water. Pour this solution into the reductant bottle and connect the reductant line from the pump. (Stannous sulfate may be substituted.)

7.3 NH_2OH-HCl (24%) - Dissolve 240 grams of hydroxylamine chloride and 240 grams sodium chloride in 2000 mL reagent water. (Sodium chloride-hydroxylamine sulfate may be used instead.)

7.4 Hydrochloric Acid (Concentrated) - J. T. Baker, "Instra-Pure"

7.4.1 Hydrochloric acid (10%) - Add 200 mL concentrated HCl to a 2 L volumetric flask containing 1 L of reagent water. Dilute to volume with reagent water.

CAUTION: Always pour acid into water.

7.5 The calibration standards are prepared by the inorganics sample preparation technician and are digested before use, following the appropriate sample preparation procedure (SPP).

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples preserved and stored according to the tables in Sample Control SOP 4.1, “Receiving Samples” and 4.6, “Storing Samples.” Sample holding times are also listed.

8.2 Aqueous samples should be preserved by acidification with nitric acid to a pH of ≤ 2 at the time of collection. Mercury samples not preserved at the time of collection with nitric acid to a pH of < 2 are preserved in-house. Samples must be preserved with nitric acid to a pH of <2, with thorough mixing, at least 24 hours prior to analysis. If only dissolved mercury is to be determined, the sample is filtered before the acid is added. For total mercury, the filtration step is omitted. Aqueous samples for all methods except ILM05.4 must be stored at room temperature or may be refrigerated at 2-6 ^{o}C.

8.3 Aqueous samples received for analysis using the ILM05.4 statement of work must be refrigerated at 2-6 ^{o}C.

8.4 For MCAWW, it is recommended that samples be sent to the lab as soon as possible after collection and acid preserved upon receipt. After acidification, the samples should be mixed, held for 16 hours, and then the pH verified to be ≤ 2. If the sample pH is > 2, repeat the process.

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8.5 Soil samples are not preserved with acid. The soil samples are stored at 2-6 ^{o}C until sample preparation.

9.0 Quality Control

9.1 Preparation Blank

9.1.1 For every 20 samples or for each digestion batch, whichever is more frequent, analyze a preparation blank. This blank is used to ascertain whether sample concentrations reflect contamination.

9.1.1.1 For CLP the blank must not contain mercury at a level above the CRQL.

9.1.1.2 For SW-846 the blank must not contain mercury at a level above the reporting limit (PQL).

9.1.1.3 For the **DoD**-QSM, the preparation blank must not contain mercury at a level above one-half the reporting limit (RL).

9.1.1.4 For MCAWW, the blank must not contain mercury at a level ≥ 10% of the analyte concentration determined in the sample or > 2.2X the analyte MDL, whichever is greater.

9.1.2 If the blank does not meet the applicable criteria above, the entire sample batch associated with the preparation blank must be re-prepared along with a new blank.

9.**1**.2.1 The only circumstance under which the preparation blank can be reported with values greater than the CRQL for CLP is when the concentration of mercury in the sample exceeds the blank concentration by a factor of 10 or more.

9.1.3 Values between the CRQL and the MDL (for CLP) or LOD and DL (for **DoD**-QSM) are reported with a "J" flag.

9.2 Matrix Spike

9.2.1 A matrix spike shall be prepared with every 20 samples or each digestion, whichever is more frequent. Samples identified as field blanks and Performance Evaluation (PE) samples, exclusive of Quarterly Blinds, shall

not be used for spiked sample analysis. For MCAWW, the matrix spike is prepared every 10 water samples.

9.2.1.1 A matrix spike duplicate is also prepared with every 20 samples for SW-846.

9.2.2 The spike recovery of the matrix spike should be within 70-130% for MCAWW and within 75-125% for CLP, and 80-120% for SW-846. The relative percent difference (RPD) between the duplicated matrix spikes should be ± 20.

9.2.2.1 If the sample concentration is less than four times the amount spiked and the recovery of the spike is less than 75% or greater than 125%, the corresponding element is flagged with an "N" on Form I to indicate that the element did not recover the matrix spike acceptably.

9.2.3 To meet the requirements of the **DoD**-QSM, the matrix spike recovery should meet the LCS control limits. The RPD between the matrix spikes should be ≤ 20%.

9.2.3.1 If the **DoD**-QSM acceptance limits are not met for the matrix spikes, contact the client for guidance.

92.3.2 If the results are reported that are associated with failing matrix spikes, indicate the specific analytes in the original sample in the narrative. Refer to the **DoD**-QSM “J” flag.

9.3 Sample Duplicate

9.3.1 A duplicate sample is prepared with every 20 samples or each digestion, whichever is more frequent. Samples identified as field blanks and Performance Evaluation (PE) samples, exclusive of Quarterly Blinds, shall not be used for duplicate sample analysis. A duplicate sample is not required for MCAWW.

9.3.2 If the sample and duplicate do not agree within 20% RPD, then the affected element is flagged with an "*" on Form I to indicate poor results duplication.

9.4 Laboratory Control Sample (LCS)

9.4.1 An LCS is prepared with every 20 samples or each digestion batch, whichever is more frequent. (An LCS is not required for CLP waters.)

9.4.2 The solid EPA LCS/ERA PPT has special control limits, which have been provided by EPA/ERA. The true value and acceptance range are subject to change without notice based upon the current lot purchased. See the certificate of analysis or the standard preparation log for the current lot in use.

9.4.3 For SW-846, in-house determined statistical limits are used. The statistical control limits are ± 20% for aqueous LCS. The solid LCS has special control limits, which have been provided by ERA. The true value is subject to change without notice based upon the current lot purchased. See the certificate of analysis or the standard preparation log for the current lot in use.

9.4.4 To meet the requirements for the **DoD**-QSM, the LCS must meet the recovery control limits of 80 - 120%.

9.4.5 To meet the requirements of the MCAWW, the LCS must meet the recovery limits of 85 - 115%. In calculating the recovery results for the MCAWW, the analyte concentration from the preparation blank must be subtracted form the LCS.

9.4.6 If the LCS fails acceptance criteria, re-analyze. If the LCS continues to fail and no instrument malfunctions or calibration failures are detected, the LCS and all associated samples and QC samples are re-digested.

9.5 CRQL Check Standard (CRI)

9.5.1 Analyze the CRI (ILM05.4) at the CRQL at the beginning of each sample analysis sequence to verify linearity near the CRQL. The CRI is repeated as the 20th sample, 40th sample, etc.

9.5.2 The acceptance criterion is ± 30% for ILM05.4.

9.6 Serial Dilution

9.6.1 For SW-846, perform a serial dilution (1:5) on one sample per analytical batch (or when a new or unusual matrix is encountered) when the analyte concentration is greater than 25 times the MDL.

9.6.2 The recovery of the analyte in the diluted sample must be within ± 10% of that in the undiluted sample.

9.6.3 If the serial dilution fails, prepare and analyze a spiked portion of the sample at 2.5 times the analyte level.

9.6.4 If the recovery is not between 85-110%, calculate the concentration of mercury in all samples in the batch by the method of standard additions. See Section 12.9.

9.7 Contingency

9.7.1 If, due to a lab accident or to QC failure, a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.7.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.7.3 Any other issues that potentially affect data quality must be addressed with the Project Manager.

10.0 Calibration and Standardization

See Section 11.0 for instrument procedures.

Note: Only one read (burn) is obtained for each analytical run.

Note: The laboratory reporting limit is at or above the lowest initial calibration standard concentration.

10.1 Initial Calibration

10.1.1 The **Leeman HYDRA AA/**PS200II must be calibrated before you can run samples. The following procedure is performed after the start-up (**Section** 11.4) and before running samples. Calibration standards are digested along with samples.

10.1.2 To perform a standard calibration, click on the WinHG icon on the desktop. The WinHG runner 1.7 screen will appear. Tighten the tubing clamps on the pump. Under the Control tab, turn pump on. The lamp should already be on, as well as the gas (0.10 LPM). Under the sample

tab, select the rack to be run. Racks are built in the Rack Editor and batches are transferred from the Sample Table on the desktop. When the Sample Table icon is clicked on, a list of sample batches appears. Click on the batch to be run and click on the Build Hg Table button at the bottom of the page. A blank table appears; press Ctrl-Shift-L and the batch will appear. Copy and paste the batch to the Rack Editor. The Client ID will be in the first columns and the laboratory ID numbers in the second column. The calibration standards will be in the far right column.

A template with the opening sequence is stored in the Rack Editor. Click on File and then open Rack 1. The continuing calibration standards can be added in the right column, where needed. Name the Rack by clicking File-Save As (usually V4mmddyy or V4mmddyA or V4mmddyB, etc.). When the rack is ready to run, select it from the drop-down list on the WinHg runner, under the Sample tab, and set the beginning and end cups. Under the Move Tip tab, press To Rinse. Click on the Run Auto button and the system will run the standards and then the samples. It is assumed that the six standards have been loaded as standards 1-6 on the standards screen.

10.1.3 The results of the calibration are automatically stored. To review the results, open the Data Base by clicking on the DB button at the top of the WinHg runner screen, then the calibration tab. The instrument generates a calibration curve based on linear regression.

10.1.3.1 The correlation coefficient must be ≥ 0.995. A forced intercept calibration model is not utilized.

10.1.3.2 For ILM05.4, standards must be within 5% of the true value (except the CRQL standard).

10.1.3.3 If above criteria are not met, check for outliers or reanalyze the entire curve. It may only be necessary to reanalyze a single outlier in order to verify the initial results. See Section 10.5.

10.2 Initial Calibration Verification/Initial Calibration Blank (ICV/ICB)

10.2.1 The ICV is digested with samples and prepared with each sample batch. The results of the ICV must agree within 10% for SW-846 and **DoD**, 5% for MCAWW, and 20% for CLP, of the "true" values. If not, the run must be terminated and the instrument recalibrated. If recalibration does not fix the problem, shut down the instrument, label with an "OUT OF CALIBRATION" tag, and request instrument service.

10.2.2 Follow the ICV with the initial calibration blank (ICB). The mercury results for the blank must be less than the EPA CRQL for CLP or less than the reporting limit (PQL) for SW-846. If not, terminate the analysis and recalibrate. A reanalysis of the ICB may be performed for SW-846 prior to recalibration. If recalibration does not fix the problem, shut down the instrument, label with an "OUT OF CALIBRATION" tag, and request instrument service.

10.2.2.1 The **DoD**-QSM and MCAWW require that the results for the ICB must be less than the MDL for MCAWW and less than or equal to the LOD for **DoD**.

10.3 Continuing Calibration Verification/Continuing Calibration Blank (CCV/CCB)

10.3.1 Verify the calibration every 10 samples and at the end of the analytical sequence using the 5.0 µg/L level calibration standard. The results of the check standard must agree within ± 20% of the true value. If not, terminate the analysis, correct the problem, and recalibrate the instrument.

10.3.1.1 For North Carolina regulatory work and for MCAWW, the check standard must agree within ± 10%.

10.3.2 Follow the CCV with the continuing calibration blank (CCB).The results for the CCB must be less than the CRQL for CLP or less than the reporting limit (PQL) for SW-846. If not, terminate the analysis and recalibrate. A reanalysis of the ICB may be performed for SW-846 prior to recalibration. If recalibration does not fix the problem, shut down the instrument, label with an "OUT OF CALIBRATION" tag, and request instrument service.

10.3.2.1 The **DoD**-QSM and the MCAWW require that the results for the CCB must be less than the MDL (for MCAWW) and less than or equal to the LOD for **DoD**.

10.4 If the calibration results are acceptable, begin running samples.

10.5 If the calibration results are unacceptable**,** rerun the standards to verify results. When an acceptable calibration is achieved, you can begin running the samples. If an acceptable calibration is not achieved upon reanalysis of the initial calibration, ICV, CCV, or CCB the entire batch must be re-digested and reanalyzed. If re-digestion and reanalysis, including all procedures in Section 11.0 regarding instrument set up and maintenance, does not yield acceptable

results, shut down the instrument, label with an "OUT OF CALIBRATION" tag, and request instrument service. After appropriate service, the entire batch must be re-digested and reanalyzed.

10.5.1 Samples bracketed by an acceptable Initial Calibration, ICV, ICB, CCV and CCB do not need to be re-digested and reanalyzed if subsequent closing CCVs/CCBs fail.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures"

Note**:** Only one read (burn) is obtained for each analytical run.

11.1 Powering up the system

11.1.1 Bring up the WinHg runner screen, go to the Control tab, and press "On," under Hg Lamp. Let the lamp warm up for at least an hour. (This should only be done if the system has been off, such as after a power failure. Otherwise the system should remain on at all times.

11.1.2 Press the "Power On" button on the back of the PS200II unit if the unit is powered off.

11.1.3 Check the reductant volume and refresh, if needed.

11.1.4 Clean the rinse tank using standard lab cleaning practices, and add fresh rinse.

11.2 Sample Analysis

Prior to analysis all samples and standards are digested following the appropriate method sample preparation procedure (SPP). The soil SOP is SPP –162, "Solid Sample Mercury Digestion by SW-846 **Methods 7471A and 7471B**." The water SOP is SPP –074, "Mercury in Water, Manual Digestion Procedure for EPA CLP and SW-846, and MCAWW."

Note: The PS200II must be calibrated before you can run samples. The calibration procedure is given in **Section** 10.0. Autosampler tables automatically go through the calibration process and proceed to run the samples.

Note: See Section 9.0 for QC samples, acceptance criteria, and corrective actions for QC failures.

11.2.1 Just before analysis, add 55 mL of DI water to all soil samples and 50 mL of reagent water to all standards. Next, add 6 mL of hydroxylamine hydrochloride solution to reduce excess permanganate, and mix until the sample is clear. The Leeman HYDRA AA/PS200II automatically adds the appropriate amount of stannous chloride during analysis. Pour standards and samples into tubes and load into autosampler racks based upon the set-up of the Rack Editor.

11.2.2 Arrange standards in increasing concentrations and calibrate instrument.

11.2.3 Analyze a solution from a different stock source than the standards for the initial calibration verification (ICV). Determine that the measured value is in agreement with the certified value. If not, recalibrate after determining the problem.

11.2.4 Analyze the calibration blank and preparation blank(s). If the calibration blank contains mercury at a value that is greater than the reporting limit terminate the analysis, correct the problem, and recalibrate. The **DoD-**QSM requires that the results for the calibration blank must be < the MDL/LOD and < ½ RL for the preparation blank.

11.2.5 Analyze the CRI standard at the beginning of each sample analysis to verify linearity near the CRQL.

11.2.6 Analyze samples, sample spike(s), and duplicate(s) monitoring quality control requirements. A serial dilution must also be analyzed for SW-846 methods. This should be a dilution of one sample within each SDG, and/or each sample matrix. This dilution is 1 in 5 (5x).

11.2.7 Verify the calibration every 10 samples and at the end of the analytical run with a CCV followed by the CCB. Samples must be bracketed by passing CCV/CCB samples. Samples associated with a failing CCV or CCB must be re-analyzed.

11.2.8 Generate the mercury run log (Attachment 4). When sample analysis is complete, the run log and original raw data is copied and placed in all associated SDG folders.

11.3 Transferring Data

11.3.1 In Database, go to the Report tab and press Generate Report. Click on Generate and the raw data will print. Highlight the PRN button and name the file as the run was named (e.g., V4mmddyy, etc.) and click Generate again. The PRN file is then used to generate the Mercury Run Log. Go to the ICP Check icon (Instrument Data Review) on the desktop and locate the PRN file from the drop-down list. Click on Mercury Run Log. The run log screen will appear and then print.

11.4 Shutdown Procedure

In routine operation, where the system is likely to be used daily, just turn the pump off under the WinHg runner control tab, move the tip To Air under the Sample tab, loosen the clamps on the pump housing, and take the lines out of the reagent vessels. For long term shut down, turn off the analyzer by flipping the switch on the back of the unit.

NOTE**:** Before shutting down the instrument, word "Idle" should appear in the field on the top of the WinHg runner screen. To interrupt or stop an ongoing procedure, press the STOP key. The instrument can then be shut down.

12.0 Data Analysis and Calculations

Calculations must be consistent with the QC SOP 13.4, "Numerical Data Reduction."

12.1 Linear Calibration using Least Squares Regression

$$y = ax + b$$

where: y = Instrument response (peak area)
a = Slope of the line (coefficient of x)
x = Concentration of the calibration standard
b = The intercept

Linear Regression by Least Squares:

$$y_1^{'} = ax_i + b$$

Where: b = intercept
x_i = Mass of the analyte in the ith calibration standard aliquot injected
$y_1^{'}$ = calculated response for the ith calibration standard

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{N}(y_i - y_1^{'})^2$$

12.2 Correlation Coefficient r:

$$r = \frac{N\left[\sum_{i=1}^{N}(x_i y_i)\right] - \left[\sum_{i=1}^{N}(x_i)\right]\left[\sum_{i=1}^{N}(y_i)\right]}{\sqrt{N\left[\sum_{i=1}^{N}(x_i^2)\right] - \left[\sum_{i=1}^{N}(x_i)\right]^2} \; x \sqrt{N\left[\sum_{i=1}^{N}(y_i^2)\right] - \left[\sum_{i=1}^{N}(y_i)\right]^2}}$$

Where: r = Correlation Coefficient
N = number of data points (equals 5 in a 5 point curve)
y = response
i = index variable (first data point i = 1)
x = Mass of the analyte in the calibration standard aliquot injected

12.3 Concentration Calculations: read the 'digestion concentration" from the calibration curve, directly from the instrument read-out or from the method of standard additions (see Section 12.10).

12.3.1 Concentration of Mercury in aqueous samples =

$$\frac{\text{(digestion concentration (µg/L)) (digestate volume (L))(dilution factor)}}{\text{(sample volume (L))}}$$

12.3.2 Concentration of Mercury in soil samples =

$$\frac{\text{(digestion concentration (µg/L)) (digestate volume (L)) (dilution factor)}}{\text{(sample weight (g)) (\% solid)}}$$

12.4 Calculation of percent recovery:

12.4.1 LCS and surrogates:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.4.2 Matrix spikes:

$$\% R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked} x\ 100$$

12.5 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.6 Calculation of RPD

$$RPD = \frac{|Value\ 1 - Value\ 2|}{(Value\ 1 + Value\ 2)/2} x100$$

12.7 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ value}}{\overline{Reference\ value}} x\ 100$$

12.8 Dilutions

12.8.1 Sample concentrations falling above the analytical range, i.e. highest calibration standard concentration, must be diluted.

12.8.2 A sample dilution must yield a value within the upper half of the analytical concentration range to be acceptable.

12.8.3 The level of dilution selected is determined by the detected concentration and the expertise of the analyst.

12.8.4 Samples are analyzed undiluted unless in the analyst's technical judgement a dilution is required based on the appearance of the sample, prior knowledge of the client or site, instrument problems related to analysis of a particular sample set, etc.

12.9 Method of Standard Additions:

To equal volumes of the sample are added a series of standard solutions containing different known quantities of mercury. All solutions are diluted to the same final volume.

Prepare addition one so that the resulting concentration is approximately 50 percent of the expected absorbance from the indigenous (from the sample matrix) analyte in the sample.

Prepare additions 2 and 3 so that the concentrations are approximately 100 and 150 percent of the expected endogenous (from the sample itself) sample absorbance.

The absorbance of each solution is determined and the result plotted on the vertical axis of a graph with the concentrations of the known standards on the horizontal axis. Extrapolate the resulting line to zero absorbance. The point of the interception with the abscissa (x-coordinate) is the endogenous concentration of the sample. See Attachment 3.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Leeman PS200 Set-up and Operation Manual"

16.2 U.S. EPA CLP SOW for Inorganic Analysis, ILM05.4

16.3 "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," SW-846, 3rd edition, Update III, Methods 7470A/7471A, Revision 1, 9/94 and Update IV, Method 7471B, Revision 2, February 2007

16.4 Methods for Chemical Analysis of Water and Wastes (MCAWW), Method 245.1, Revision 3.0, 1994

16.5 Quality Control SOP, 13.6, "Proper Documentation Procedures"

16.6 Quality Control SOP 13.4, "Numerical Data Reduction"

16.7 NELAC Standards, effective, July 2003, plus revisions

16.8 **QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007**.

16.9 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.10 Code of Federal Register, 40 CFR, Part 136, "Guidelines for Establishing Test Procedures for Priority Pollutants"

16.11 CompuChem Quality Manual, Revision 13, November 17, 2009

16.12 Sample Control SOP 4.6, "Storing Samples"

16.13 Department of Defense Quality Systems Manual of Environmental Laboratories, Version 4.**2**, **10**/**25**/**2010**

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – Example Standards/QC Preparation Log for Cold Vapor Mercury

17.2 Attachment 2 – Example Mercury Run Log

17.3 Attachment 3 – Standard Addition Plot from SW-846 7000B

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Attachment 1

Standards/QC Preparation Log for ICP Metals/Mercury Page 1 of 3 **Case/SDG:**_____
COMPUCHEM a division of Liberty Analytical Corp. Worksheet 1Q (1) 2

REAGENTS	REF#[1]	MANUFACTURER	LOT#	EXPIRATION DATE
SULFURIC ACID (H_2SO_4)	0C02009	CALEDON	68764	10/12
NITRIC ACID (HNO_3)	0B02020	JT BAKER	H20053	02/13
POTASSIUM PERMANGANATE ($KMnO_4$)	9L30017	JT BAKER	H32614	12/12
HYDROCHLORIC ACID (HCL)	9L29009	CALEDON	71900	12/12
POTASSIUM PERSULFATE ($K_2S_2O_8$)	0C02011	JT BAKER	H02H06	05/12
HYDROGEN PEROXIDE (H_2O_2)	0C02013	CALEDON	69862	10/10
STANDARDS				
XCL 20R	0B26007	HIGH PURITY	0925409	09/11/10
XCL LCS 10XR	9L15003	HIGH PURITY	0935232	12/10/10
ERA PPT (540)	0C01027	ERA	540	12/03/10
EPA LCS (0405)	0B26009	EPA	0405	08/05/10
ICV-1 (0307)	0B04003	EPA	0307	03/12/11
ILM05.3 MATRIX SPIKE	9L15006	HIGH PURITY	0916332	06/15/10
SILVER (Ag) 100ppm	9L15009	HIGH PURITY	0934323	12/15/10
TCLP 500	9L21002	HIGH PURITY	0934202	12/10/10
ARSENIC (As) 1000ppm	9F17001	HIGH PURITY	0911221	12/05/10
MERCURY (Hg) 20ppm	9L15007	HIGH PURITY	0916320	06/15/10
ICV-5 (0508) Hg	0C01026	EPA	0508	09/15/10

[1]Ref#=Logbook ID, Page number, and item number from Materials Receipt Log
[2]Standard Lot Number consist of standard ID, date prepared, and initials of preparer

STANDARD SOLUTIONS PREPARED BY:_____ DATE:_____

ENTERED BY:_____ DATE:_____

REVIEWED BY:_____ DATE:_____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this worksheet attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

12/03/07:RM

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Attachment 1 (Continued)

Standards/QC Preparation Log for ICP Metals/Mercury Page 2 of 3 **Case/SDG:**_____

COMPUCHEM a division of Liberty Analytical Corp. Worksheet 1Q (1) 2

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS
Hg 100ppb-03-15-10-DD	Place in a 100mL volumetric flask: 0.5mL of 20ppm Hg stock solution 2.5mL of concentrated HNO_3 Baker instra-analyzed Trace Metals grade or equivalent Dilute to 100mL volume with reagent water (**PREPARE DAILY**)
$K_2S_2O_8$- 03-15-10-DD Potassium Persulfate	Place in a 1000mL Erlenmeyer to a re-pipettor bottle (**PREPARE AS NEEDED**)
$KmnO_4$. 03-15-10 –DD Potassium Permanganate	Place in a 2000mL volumetric flask: 100.0g of $KmnO_4$ Baker instra-analyzed Trace Metals grade or equivalent Dilute to 2000mL with reagent water (**PREARE AS NEEDED**)
HNO_3- 03-09-10 - JR Nitric Acid	Place in a 2000mL volumetric flask: 500mL of Deionized water 1000mL of concentrated HNO_3 Baker instra-analyzed Trace Metals grade or equivalent Dilute to 2000mL with reagent water (**PREPARE AS NEEDED**)
HCl- 02-12-10 - JR Hydrochloric Acid	Place in a 2000mL volumetric flask: 500mL of Deionized water 1000mL of concentrated HCl Baker instra-analyzed Trace Metals grade or equivalent Dilute to 2000mL with reagent water (**PREPARE AS NEEDED**)
Aqua Regia- 02-10-10 - DD	Place in a 1000mL container: 600mL of Concentrated HCL 200mL of Concentrated HNO3 **(PREPARE AS NEEDED)**

[1]Ref#=Logbook ID, Page number, and item number from Materials Receipt Log
[2]Standard Lot Number consist of standard ID, date prepared, and initials of preparer

STANDARD SOLUTIONS PREPARED BY:_____ DATE:_____

ENTERED BY:_____ DATE:_____

REVIEWED BY:_____ DATE:_____

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12/03/07:RM

REVIEWED BY:_____ DATE:_____

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Attachment 1 (Continued)

Standards/QC Preparation Log for ICP Metals/Mercury Page 3 of 3 **Case/SDG:**_____
COMPUCHEM a division of Liberty Analytical Corp. Worksheet 1Q (1) 2

STANDARD LOT NUMBER[2]	PREPARATION INSTRUCTIONS	Resultant Analyte Concentration
ICV-5- 03-15-10-DD	Pipet into a 300mL BOD Bottle 1.0mL of ICV-5 and 99mL of reagent water. Digest according to sample preparation instructions	4.0µg/L
S0/ICB/CCB- 03-15-10-DD	Pipet into a 300mL BOD Bottle 100mL of reagent water Digest according to sample preparation instructions	0.0µg/L
S0.2/CRA/CRI- 03-15-10-DD	Pipet into a 300mL BOD Bottle 0.2mL of Hg 100ppb and 99.8mL of reagent water Digest according to sample preparation instructions	0.2µg/L
S0.5- 03-15-10-DD	Pipet into a 300mL BOD Bottle 0.5mL of Hg 100ppb and 99.5mL of reagent water Digest according to sample preparation instructions	0.5µg/L
S1.0- 03-15-10-DD	Pipet into a 300mL BOD Bottle 1.0mL of Hg 100ppb and 99mL of reagent water Digest according to sample preparation instructions	1.0µg/L
S5.0/CCV- 03-15-10-DD	Pipet into a 300mL BOD Bottle 5.0mL of Hg 100ppb and 95mL of reagent water Digest according to sample preparation instructions	5.0µg/L
S10.0- 03-15-10-DD	Pipet into a 300mL BOD Bottle 10mL of Hg 100ppb and 90mL of reagent water Digest according to sample preparation instructions	10.0µg/L

[1]Ref#=Logbook ID, Page number, and item number from Materials Receipt Log
[2]Standard Lot Number consist of standard ID, date prepared, and initials of preparer

STANDARD SOLUTIONS PREPARED BY:_____ DATE:_____

ENTERED BY:_____ DATE:_____

REVIEWED BY:_____ DATE:_____

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Attachment 2

Maintenance: _____

Method(s) (circle one): **ILM04.1** **ILM05.4** **Others** 7470A 7471A

Operator(s): _____ **Date/Time** _____ **to** _____

Printed: 5/14/2009

↑	Specified element(s) fail(s) high
↓	Specified element(s) fail(s) low
RR	Re-run sample(s) neat
RR #X	Re-run at a #-fold dilution
#X	Sample run at a #-fold dilution (aliquot of sample diluted with acidified water)
L	Matrix = leachate
W	Matrix = water
S	Matrix = solid
B	Matrix = biological tissue

SDG	Matrix	Comments

Batch

HG Run Log

File Name:

Cup	Sample ID	Client ID	Sample Type	DF	Date	Time	Results	Comments

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Reviewed By: _____ Date: _____ Page 1 of 1

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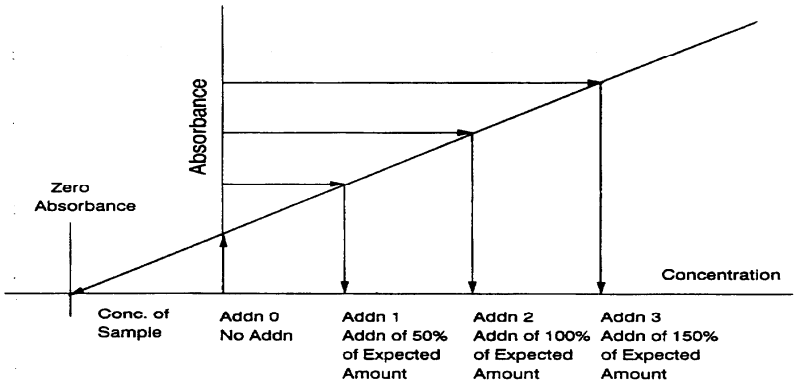
Attachment 3

FIGURE 1

STANDARD ADDITION PLOT



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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-1051 SOP Section #: 3.4.4 Revision #: 8

SOP Title: Reactive (Total Releasable) Cyanide by SW-846

Effective date: (QA fills in) 06-06-11

- Procedure prepared by: Judith Dozier Date: 06-06-11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) JCS Date: 06/06/11
- Reason for change: annual review
- This procedure meets the requirements of the following approved method references: SW846, 3rd Edition, Update III, 12/96 Chapter 7.3.3 and Method 9014; Depart. of Defense Quality Systems Manual for Environmental Laboratories Sec. 4.2, October 25, 2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -1051: Reactive (Total Releasable) Cyanide by SW-846

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Sample Preparation Procedure -1051: Reactive (Total Releasable) Cyanide by SW-846

1.0 Scope and Application

This standard operating procedure (SOP) describes the process to determine the specific rate of release of hydrocyanic acid (HCN) upon contact with an aqueous acid. This method is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures. This method measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

The current EPA guidance level for total releasable cyanide is 250 mg HCN/Kg of waste. The reporting limit is 125 mg/Kg. Because the regulatory level and reporting limit are so high, a method detection limit (MDL) is not required to be determined.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

An aliquot of acid is added to a fixed weight of waste in a closed system. The generated gas is swept into a scrubber and the analyte quantitated.

3.0 Definitions

3.1 Reactivity - According to the regulation in 40 CFR 261.23, a waste exhibits the characteristic of reactivity if a representative sample of the waste has any of the following properties:

- It is normally unstable and readily undergoes violent change without detonating.
- It reacts violently with water.

- It forms potentially explosive mixtures with water.

- When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

- It is a cyanide- or sulfide-bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.

- It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.

- It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.

- It is a forbidden explosive, as defined in 49 CFR 173.51, or a Class A explosive, as defined in 49 CFR 173.53, or a Class B explosive, as defined in 49 CFR 173.88.

- This definition is intended to identify wastes that, because of their extreme instability and tendency to react violently or explode, pose a problem at all stages of the waste management process.

3.2 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.3 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.4 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99%

confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.5 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.6 Reporting Limit (RL) –

3.6.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration.

3.6.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported between the DL and the LOQ must be flagged as estimated values.

3.**7** Reporting Limit – The laboratory reporting limit is one half the current EPA guidance level regulatory reporting limit.

3.**8** Reporting Units – mg/Kg

3.**9** A **Sample Delivery Group (**SDG**)** is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must

also be prepared together at a rate of 5% for DoD and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.10 DoD-QSM – Department of Defense Quality Systems Manual

3.11 SC DHEC – South Carolina Department of Health and Environmental Control

3.12 NCDENR – North Carolina Department of the Environment and Natural Resources

3.13 Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.14 LIMS – Laboratory Information Management System

4.0 Interferences

Interferences are undetermined.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. Testing must be performed in a ventilated hood. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff members are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory. The MSDS are located in the Quality Assurance department.

6.0 Equipment **and** Supplies

6.1 Distillation Equipment

6.1.1 500 mL round bottom flask

6.1.2 Calibrated 50 mL scrubber

6.1.3 500 mL side arm

6.1.4 Nitrogen gas with rotometer

6.1.5 Round bottom flask stirrer

6.2 Ordinary laboratory glassware

6.2.1 10 mL class A microburet

6.2.2 Magnetic stirrer, stir bars, pipets, standard lab equipment

6.2.3 500 mL beakers

6.2.4 Graduated specimen cups with at least 100 mL capacity

6.3 Analytical balance, capable of weighing to nearest 0.01 g

6.4 Element DataSystem® LIMS computer software by Promium

7.0 Reagents

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted. All standards and reagents are subject to change in vendor and in concentration.

Remember to record all reagents and standards in the Standards/QC Preparation Log (Attachment 2) or in LIMS. Refer to the Standards Preparation Worksheets or Promium Element LIMS and applicable SOPs for details on preparation and traceability of standards used in this procedure.

Note: All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date five years from receipt. All lab prepared regents must be assigned an expiration date one year from preparation.

7.1 Reagent water - All water used during preparation must be reagent-grade Type I with regard to resistivity of >10 megohm-cm (20th Edition of Standards Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Sulfuric acid (0.1N) H_2SO_4

7.2.1 Add 2.8 mL of concentrated H_2SO_4 DI water and dilute to 1 liter.

7.3 Sulfuric Acid (0.01N) H_2SO_4

7.3.1 Add 100 mL of 0.1N H_2SO_4 to DI water and dilute to 1 liter

7.4 Standard silver nitrate titrant, 0.0192N

7.4.1 Purchased commercially prepared and certified

7.5 Rhodanine Indicator

7.5.1 Add 20 mg p-Dimethylaminobenzalrhodanine to 100 mL of acetone.

7.6 Cyanide reference solution (KCN) **– 1,000 ppm Standard purchased from NSI or equivalent supplier**

NOTE: For this procedure 1 mL of cyanide solution is equivalent to 1 mg. All results are reported in mg/Kg.

7.7 Sodium hydroxide solution (0.25N NaOH)

7.7.1 Dilute 10 g of NaOH pellets in 1 liter DI H_2O.

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples must be stored refrigerated at 4 °C ± 2 °C and run as soon as possible but within 7 days of sampling. The holding time for this test is 7 days from date of collection.

8.3 It is suggested that samples of cyanide wastes be tested as quickly as possible. Although they can be preserved by adjusting the sample pH to 12 with strong base, this will cause dilution of the sample, increase the ionic strength, and, possibly, change other physical or chemical characteristics of the waste which may affect the rate of release of the hydrocyanic acid. Storage of samples must be under refrigeration in the dark.

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9.0 Quality Control

The following quality control samples are required for this analysis:

9.1 Method Blank (Preparation or Reagent Blank): A reagent blank is used to establish that the reagents are free from contamination. One method blank must be analyzed per every preparation batch of up to 20 samples. For SC DHEC, analyze the blank every 10 samples.

9.1.1 If any value present in the method blank exceeds the RL (reporting limit), the entire batch must be reprocessed using clean glassware.

9.1.2 For DoD-QSM ff any value present in the method blank exceeds the ½ RL, the entire batch must be reprocessed using clean glassware.

9.2 Laboratory Control Sample (LCS): The LCS solution is 10 mL of cyanide reference solution. The LCS must be prepared with every batch of up to 20 samples. For SC DHEC, analyze the LCS every 10 samples.

9.2.1 The accuracy of the process is determined from the LCS recovery. Statistical control limits for the LCS are 14 - 77% of the true value (**1000** mg/Kg).

Disclaimer: The preparation procedure for reactive cyanide calls for the use of a normality of sulfuric acid, which is not amenable to releasing the cyanide effectively. Based on information obtained from the RCRA Hotline, the expected cyanide recoveries using the method as written range between 5 -10%.

9.3 A duplicate sample must be prepared with every batch of 20 samples for NCDENR. Results must agree within ± 20%. For SC DHEC a duplicate is required every 10 samples.

9.4 Contingency

9.4.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

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9.4.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.4.3 Any other issues that potentially effect data quality must be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 Balance Calibration

10.1.1 Ensure the balance is calibrated for the day before use. Refer to Quality Control SOP 13.16, "Top-Loading Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in QC SOP **13.6**: Proper Documentation Procedure.

Record all information in the Reactive Cyanide Preparation and Run Log (Attachment 2). Include all pertinent information such as sample size, dilution factors, date(s) of analysis, and sample ID. As the analysis proceeds, discrepancies or deviations must be written in the logbook immediately.

11.1 Preparation Procedure

11.1.1 Add 50 mL of 0.25N NaOH solution to a calibrated scrubber.

11.1.2 Close the system and adjust the flow rate of nitrogen gas, using a rotometer. The flow rate should be approximately 60 mL/minute.

11.1.3 Mix the sample well, discard any foreign objects such as sticks, stones/rocks, and leaves. Weigh 10.0 g or measure 10 mL of the waste to be tested and add to a 500 mL round bottom flask with stir bar. Use as little water as possible to transfer soil into flask.

11.1.4 With the nitrogen flowing, add 250 mL of 0.01N $H_2SO_4$to the flask. Start the 30 minute test period.

11.1.5 Begin stirring while the acid is entering the flask. The stir speed must remain constant throughout the test.

Note: Stirring should not create a vortex.

11.1.6 After 30 minutes, close off the nitrogen flow and disconnect the scrubber. Let air purge the system for approximately 2 minutes.

11.1.7 Drain the solution from the scrubber into a graduated plastic specimen cup and dilute to approximately 100 mL if necessary. The scrubber volume is used for testing both reactive cyanide and reactive sulfide from one scrubber.

11.1.8 For the LCS, drain the solution from the scrubber and dilute to 100 mL if necessary.

11.2 Titration Procedure

11.2.1 Add 20 mL of sample to 100 mL of DI water in a beaker. Add approximately 1.0 mL of Rhodanine indicator. For the LCS use 50 mL.

11.2.2 Titrate with 0.0192N $AgNO_3$ to the first change in color from yellow to brown-pink. The titration must be performed slowly with constant stirring. Record titrant volume in the reactive cyanide logbook (attachment 2).

Note: If needed to become familiar with the endpoint of the titration and the amount of indicator to be used before actually titrating the sample, titrate a water blank using the same amount of sodium hydroxide and indicator as the sample.

The titration is based on the following reaction:

$$Ag^+ + 2CN^- \rightarrow [Ag\,(CN^-)_2]$$

When all cyanide has complexed and more silver nitrate is added, the excess silver combines with the rhodanine indicator to turn the solution yellow and then brownish-pink.

12.0 Data Analysis and Calculation

All calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Reactive Cyanide

12.1.1 Calculate concentration of CN in the scrubber as follows:

$$CN^{-}\ (ug/l) = \frac{(A-B)}{C}\ x\ D\ x\ \frac{2\,mole\,CN-}{1\,eq,\,AgNO_3}\ x\ \frac{26.02\,g\,CN-}{1\,mole\,CN-}\ x\ \frac{1\,x\,10^6\ \mu g}{1\,g}$$

where: A = mL of $AgNO_3$ for titration of sample
B = mL of $AgNO_3$ for titration of blank
C = mL of sample titrated (20, or the actual volume taken from the scrubber, if not 20)
D = actual normality of $AgNO_3$ (0.0192N)

12.1.2 Calculate the specific rate of release of HCN using the following equation:

$$R = \text{Specific Rate of Release (mg/kg/sec.)} = \frac{X\ x\ L}{W\ x\ S}$$

where: X = CN (mg/L) = CN- (µg/L) ÷ 1000
L = Total volume of solution in scrubber (L)
W = Weight of waste used (Kg)
S = Time of measurement (sec.)=Time N stopped-Time N started

12.1.3 Calculate the total releasable HCN (mg/kg) using the following equation:

Total releasable HCN (mg/kg) = R x S

12.2 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values

x_i = each individual value used to calculate the mean
x = the mean of n

12.3 Calculation of the standard deviation of a set of values:

$$Standard\ deviation = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.4 Calculation of percent recovery:

12.4.1 LCS:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.4.2 Matrix spikes:

$$\%\ R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\ 100$$

12.5 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.6 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{\left(Value\ 1\ +\ Value\ 2\right)/2}\ x100$$

12.7 Calculation of %Difference (%D)

$$\%Diff = \frac{Value\ -\ \overline{Reference\ \ value}}{\overline{Reference\ \ value}}\ x\ 100$$

12.8 Data Review and Verification

12.8.1 Review and verification must follow the procedures in the Wet Chemistry and Organic Characterization SOP 14.3.2.1, "Data Management: Wet Chemistry Data Review and Verification"

12.8.2 Initial review is performed by the analyst who also generates the final report. A peer or supervisory review is performed prior to release of analytical results to the client.

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods For Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 3rd Edition, Update III, 12/96, Chapter 7.3.3 and Method 9014

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 QC SOP **13.6**: Proper Documentation Procedures

16.4 QC SOP **13.4**: Numerical Data Reduction

16.5 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.6 Hazardous Waste Management **and** Safety SOPs: **12.1** "Hazardous Waste Disposal" and **12.2** "Spill Control **and** Cleanup."

16.7 NELAC Standards, effective July 2003, plus revisions

16.8 **EPA QA/G6: Guidance for Preparing of Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007**.

16.9 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.10 Sample Control SOP 4.1, "Receiving Samples"

16.11 Sample Control SOP 4.6, "Storing Samples"

16.12 Quality Control SOP 13.16, "Top-Loading Balance Calibration and Maintenance."

16.13 Inorganic Wet Chemistry and Organic Characterization SOP 14.3.2.1, "Data Management: Wet Chemistry Data Review and Verification"

16.14 DoD Quality Systems Manual for Environmental Laboratories, Version 4.**2**, **October 25, 2010**, plus revisions

17.0 Attachments as Tables, Diagrams **and** Flowcharts

17.1 Attachment **1** – **Example** Reactive Cyanide Preparation and Analysis **Worksheet**

17.2 Attachment **1** – **Example** Reagent/QC Preparation for Reactive Cyanide Analysis **Worksheet**

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Attachment 1

CompuChem
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Reactive Cyanide Preparation and Analysis
Method SW846 Chapter 7/9014

Date: _____/Time: _____

Worksheet 1 B(5) Rev. 2
Analyst No.: _____

SDG: _____
Batch No.: _____

Cond	CompuChem No.	Sample Type	A Initial Sample (kg)	B Total Scrubber Vol. (L)	C Scrubber Vol. Titrated (mL)	D Titrant Vol. (mL)	**Results** Total Releasable HCN in water/soil (mg/kg)	Comments
	Titration Blank	TBS	NA	NA	NA	0.10	NA	D1 = TBS value in column D
			0.01	0.05	20		-24.9792	reaction time = 30 minutes

Reactive Time
Start: _____
Finish: _____

Duplicates ± 20%	%RPD=		LCS Range 14-77%*	% Rec =	
			*Based upon information obtained from the RCRA Hotline, the expected cyanide recoveries using this method is between 5-10%.		

Total Releasable HCN, mg/kg = $\frac{(D - D1)}{C} \times 0.0192N\ AgNO_3 \times \frac{1eq.\ AgNO_3}{1\ L} \times \frac{2\ moles\ CN^-}{1eq.\ AgNO_3} \times \frac{26.03g\ CN^-}{1\ mole\ CN^-} \times \frac{1000mg}{1g} \times \frac{B}{A} = (D - D1) \times 249.7920$**
**when B=0.05 & C=20 & A=0.01

Reviewed By: _____ Date: _____

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

6/3/11:jad

Note: Attachment subject to change without notice.

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Attachment 2

Reagent/QC Preparation for Reactive Cyanide Analysis Seq. File Name:_____

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 L(8) Rev. 2 Page 1 of 1

REAGENTS*	REF#	Manufacturer	Lot#	Expiration Date
Reactive Cyanide Reference Solution				
Sulfuric Acid (H_2SO_4)				
Sodium Hydroxide (NaOH)				
Silver Nitrate				
Rhodanine				

REAGENT/STANDARD LOT NUMBER**	PREPARATION INSTRUCTIONS
0.1N H_2SO_4-	Add 2.8 mL concentrated H_2SO_4 to reagent water and dilute to 1 L.
0.01N H_2SO_4-	Add 100 mL of 0.1N H_2SO_4 to reagent water and dilute to1 L.
Reactive Cyanide reference solution (LCS MS/MSD) 1,000 mg/kg -	Pipet 10 mLs of the Cyanide Reference Solution into a round bottom flask. Prepare fresh with each analysis
Rhodanine Indicator -	Add 20 mg of p-Dimethylaminobenzalrhodanine to 100 mL of acetone
0.25N NaOH-	Dissolve 20 g of NaOH pellets in 2 liters of reagent water.

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log **Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

Reagent/Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

6/3/11:jad/v

Note: Attachment subject to change without notice.

SOP 3.5.9.1

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 567 SOP Section #: 3.5.9.1 Revision #: 6

SOP Title: Ignitability by SW-846 Method 1010A and ASTM D93-80 (Pensky-Martens)

Effective date: (QA fills in) 03-23-2010

- Procedure prepared by: [signature] Date: 03-18-2010
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Rebecca K Mead Date: 3/23/10
- Reason for change: Update Method to 1010A
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Ed, Update III B, 11/04, Method 1010A and Chapter 7; ASTM D93-80, "Flash Point by Pensky-Martens Closed Tester, IP-34/75

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: Bob Meiner Date: 06-14-2011

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 567: Ignitability by SW-846 Method **1010A** and ASTM D93-**80** (Pensky-Martens)

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Instrument Procedure 567: Ignitability by SW-846 Method 1010 and ASTM D93-~~00~~ 80 (Pensky-Martens)

JY 02/04/11

1.0 Scope and Application

The objective of this ignitability characteristic **procedure** is to identify wastes that either present fire hazards under routine storage, disposal, and transportation or are capable of severely exacerbating a fire once started. A solid waste exhibits the characteristic of ignitability if a representative liquid sample of the waste has a flash point < 60° C (140° F). This method uses **a** Pensky-Martens Closed Cup Tester to determine the flash point of liquids, including those that tend to form a surface film under test conditions. Liquids containing non-filterable, suspended solids or high in viscosity can also be tested using this method.

Method detection limits (MDLs) are not appropriate for this method. The cut-off point for this procedure is 140° F.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary Method

The sample is heated at a slow, steady rate with continual stirring. A small flame is directed into the cup at regular intervals with simultaneous interruption of stirring. The flash point is the lowest temperature at which application of the test flame ignites the vapor above the sample.

3.0 Definitions

3.1 Flash point - The lowest temperature corrected to a barometric pressure of 101.3 Kpa (760 mm Hg) at which application of a test flame causes the vapor of a specimen to ignite under specific test conditions.

Occasionally, the application of the test flame will cause a blue halo or an enlarged flame. This generally occurs near the actual flash point but in some cases, especially with halogenated hydrocarbons and mixtures, it can occur at any temperature. These phenomena are not to be considered true flash points.

3.2 Reporting Units – degrees Fahrenheit

3.3 An SDG is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.4 SC DHEC – South Carolina Department of Health and Environmental Control

3.5 DoD-QSM – Department of Defense Quality Systems Manual

3.6 ASTM - American Society for Testing and Materials

3.7 Highly viscous material - apparent viscosity equal to or greater than 30 weight motor oil

4.0 Interferences

4.1 Erroneously high flash points may be obtained if precautions are not taken to avoid the loss of volatile material. Do not open containers unnecessarily and make a transfer unless the sample temperature is at least the equivalent of 8° C (18° F) below the expected flash point. Do not use samples from leaky containers for these test methods.

4.2 Samples containing dissolved or free water may be dehydrated with calcium chloride or by filtering through a qualitative filter paper or a loose plug of dry absorbent cotton. Warming the sample is permitted, but it must not be heated for

prolonged periods or above a temperature of 17° C (30° F) below its expected flash point. **Samples suspected of containing volatile material should not be preheated.**

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.3 Flash point can indicate the possible presence of highly volatile and flammable materials in a relatively nonvolatile or nonflammable material. For example, an abnormally low flash point on a sample of kerosene can indicate gasoline contamination.

5.4 The operator must exercise and take appropriate safety precautions during the initial application of the test flame, since samples containing low-flash material can give an abnormally strong flash when the test flame is first applied.

5.5 **WARNING**: P-xylene and chlorobenzene are extremely flammable and harmful if inhaled. Keep away from heat, sparks, and open flame. Keep containers closed. Use with adequate ventilation. Avoid build-up of vapors and eliminate all sources of ignition, especially non-explosion proof electrical apparatus and heaters. Avoid breathing vapor or spray mist. Avoid prolonged or repeated contact with the skin.

6.0 Equipment & Supplies

6.1 Pensky-Martens Closed Flash Tester, equipped with an ASTM 9F Pensky-Martens low range thermometer having a range from 20° to 230° F (-5°-110° C)

6.2 Nova Mercury Barometer, or equivalent

7.0 Reagents and Standards

The preparation of all standards is documented in the Run/Standard Log (Attachment 1).

Note: **All reagents and standards must be ACS (American Chemical Society) grade or equivalent.** All standards are subject to change in vendor and in concentration.

7.1 Reagent Water-All water used during preparation must be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (20th Edition of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 EM Science certified p-xylene, reagent grade, Class 1C purity 99.8%, or equivalent, flash point 81° F

7.3 J.T. Baker certified chlorobenzene (mono), reagent grade, Class 1C, or equivalent, flash point 82° F

7.4 30 weight motor oil

8.0 Sample Collection, Preservation, & Storage

8.1 Samples are collected, preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples must not be collected or stored in plastic containers, since volatile material may diffused through the walls of the **container**.

8.3 Prior to analysis, all samples must be stored under refrigeration at $4^{o} \pm 2^{o}$ C in the wall-in storage unit. After analysis, samples are returned to Sample Control for long-term storage and disposal.

9.0 Quality Control

9.1 Laboratory Control Sample

9.1.1 For each sample batch of up to 20 samples, a Laboratory Control Sample (LCS), or blank spike (BS), must be analyzed. The LCS is prepared using p-xylene and analyzed as a liquid sample.

9.1.2 The LCS flash point must be 81° ± 2° F. If the LCS is outside of this range the entire batch must be reanalyzed.

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9.2 Duplicates

9.2.1 For each sample batch of up to 20 samples, a duplicate must be analyzed. The duplicate frequency for SC DHEC is one in 10 samples.

9.2.2 If the duplicate and the sample do not agree **within the following temperature ranges** the duplicate and the sample must be reanalyzed**:**

9.2.2.1 ± 4° F for liquids

9.2.2.2 ± 9° F for solid materials and highly viscous materials

9.3 Contingency

9.3.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.3.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.3.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.3.4 Any other issues that potentially effect data quality must also be addressed with the Project Manager.

10.0 Calibration & Standardization

10.1 Determine the flash point of chlorobenzene, following the directions in section 11.1. Note the WARNING in the Safety section above. Record the flash point on the run log (Attachment 1).

10.2 If the flash point obtained for chlorobenzene is not within the limits (82° ± 2° F), check the condition and operation of the apparatus, especially the tightness of the lid, the action of the shutter, and the position of the test flame. Repeat the test until criteria are met.

11.0 Procedure

Documentation must follow the requirements in QC SOP: Proper Documentation Procedures. Record all information on the Ignitability Run Log (Attachment 1).

11.1 Determination of Flash Point of Ordinary Liquids

11.1.1 Record the barometric pressure in mm Hg (millimeters of mercury)

11.1.**2** Thoroughly clean and dry the cup and its accessories before starting the test. Be sure to remove any solvent that has been used to clean the apparatus.

11.1.**3** Fill the cup with the sample to be tested to the level indicated by the fill line in the cup.

11.1.**4** Place the cup in the heating apparatus and assemble the top and lock it into place. Insert the thermometer.

11.1.**5** Bring the material to be tested and the tester to a temperature of 60° F (± 10° F) or **to 30**° F lower than the estimated flash point, whichever is lower.

11.1.**6** Light the test flame and adjust it to 4 mm ($^{5}/_{32}$ in.) in diameter.

11.1.**7** Turn on the heating apparatus and adjust the rate of temperature increase to approximately 9-11°F/minute. Apply continuous stirring; making sure that it is stirring in a downward direction.

11.1.**8** Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is gently but quickly lowered into the vapor space of the cup. Leave the flame in this position for one second, and quickly return it to its original starting position.

NOTE: Do not stir the sample while applying the test flame.

11.1.**8.1** Repeat this procedure for every 2° F in temperature increase until either the flash point is observed, or the cut-off point of 140° F is reached.

11.1.8.2 Record the flashpoint to the nearest 1° F

11.1.8.3 Apply the correction calculation for the flash point based on the barometric pressure as described in Section 12.3. Record the corrected flashpoint to the nearest 1° F.

11.2 Determination of Flash Point of Suspensions of Solids and Highly Viscous Materials

11.2.1 Record the barometric pressure in mm Hg (millimeters of mercury)

11.2.2 Thoroughly clean and dry the cup and its accessories before starting the test. Be sure to remove any solvent that has been used to clean the apparatus.

11.2.3 Fill the cup with the sample to be tested to the level indicated by the fill line in the cup.

11.2.4 Place the cup in the heating apparatus and assemble the top and lock it into place. Insert the thermometer.

11.2.5 Bring the material to be tested and the tester to a temperature of 60° F (± 10° F) or to 30° F lower than the estimated flash point, whichever is lower.

11.2.5 Light the test flame and adjust it to 4 mm ($^{5}/_{32}$ in.) in diameter.

11.2.6 Turn on the heating apparatus and adjust the rate of temperature increase to approximately 2-3 °F/minute throughout the duration of the test. Apply continuous stirring; making sure that it is stirring in a downward direction.

11.2.7 Apply the test flame by operating the mechanism on the cover which controls the shutter and test flame burner so that the flame is gently but quickly lowered into the vapor space of the cup. Leave the flame in this position for one second, and quickly return it to its original starting position.

11.2.7.1 Repeat this procedure for every 2° F in temperature increase until either the flash point is observed, or the cut-off point of 140° F is reached.

11.2.7.2 Record the flashpoint to the nearest 1° F

11.2.7.3 Apply the correction calculation for the flash point based on the barometric pressure as described in Section 12.3. Record the corrected flashpoint to the nearest 1° F.

12.0 Data Analysis & Calculations

Calculations must be consistent with the QC SOP: Numerical Data Reduction.

12.1 Calculation of percent recovery for ICV and LCS:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\ 100$$

12.2 Calculation of %Difference (%D)

$$\%Diff = \frac{Value - \overline{Reference\ value}}{\overline{Reference\ value}}\ x\ 100$$

12.3 Calculation for corrected flash point

12.3.1 Observe and record the ambient barometric pressure at the time of the test. When the pressure differs from 101.3 Kpa (760 mm Hg), correct the flash point as follows:

Corrected flash point = F + 0.06 (760 - P)

where: F = observed flash point, degrees F
P = ambient barometric pressure, mm Hg

12.3.2 Record the corrected flash point to the nearest 1° F.

13.0 Method Performance

This method was validated through in-house laboratory studies of precision and accuracy for single analyst. (The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods **for** Evaluating Solid Waste, Physical/ Chemical Methods, SW-846, 3rd Edition, **Update IIIB, 11/04, Method 1010A and Chapter 7.**

16.2 **ASTM** D93-**80, "Flash Point by Pensky-Martens Closed Tester,"** IP-34/**75.**

16.3 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.4 ~~QC SOP:~~ QC SOP 13.6: "Proper Documentation Procedures"

16.5 ~~QC SOP:~~ QC SOP 13.4: "Numerical Data Reduction"

06-14-2011

16.6 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.7 Hazardous Waste Management & Safety SOPs: "Hazardous Waste Disposal" and "Spill Control & Cleanup."

16.8 NELAC Standards, effective July 2003, plus revisions

16.9 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.10 CompuChem Quality Manual, Revision ~~13, November 17, 2009~~ 14, January 25, 2011, plus revisions 06-14-2011

16.11 Sample Control SOP 4.1, "Receiving Samples"

16.12 Sample Control SOP 4.6, "Storing Samples" 06-14-2011

16.13 DoD QSM Version 4.2, 10/25/2011

17.0 Attachments as Tables, Diagrams, Flowcharts & Validation Data

17.1 Attachment 1 - Ignitability Run/Standards Log

Attachment 1

CompuChem, a division of Liberty Analytical Corp.

Ignitability - Method 1010A

Worksheet 1 T(6) SDG: _____ Page __1___ of __1___

Analyst No.: _____ Date: _____

Ambient Barometric Pressure (mm Hg): _____ Batch No.: _____

Cond	CompuChem No.	Sample Type	Observed Flash Point (F)	Corrected Flash Point (F) Results	Comments
				45.6	

Corrected Flashpoint = F + 0.06(760-P)	where:	F = observed flashpoint, degrees F	
Method Upper Range = 140°F		P = ambient barametric pressure, mm Hg	

Ref. No. Chlorobenzene =		Ref. No. p-Xylene=	
Chlorobenzene TV =	82 ± 2°F	p-Xylene TV =	81 ± 2°F

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

03/22/10:rkm

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Note: Attachment subject to change without notice.

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501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP575 SOP Section #: 3.5.14.2 Revision #: 6

SOP Title: Corrosivity Characterization by pH Determination in SW-846

Effective date: (QA fills in) 6/29/11

- Procedure prepared by: Judith Dozier Date: 6/28/11
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Meiere Date: 06-29-2011
- Reason for change: addition of Method 9040C; addition of LIMS information
- This procedure meets the requirements of the following approved method references: Test Method for Evaluating Solid Wastes; Physical/Chemical Methods, SW-846, 3rd Ed Update IIB, 1/95, Method 9040B, Update III B, 11/04, Method 9040C; DoD QSM Version 4.2, 10/25/10

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 575: Corrosivity Characterization by pH Determination in SW-846

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Instrument Procedure 575: Corrosivity Characterization by pH Determination in SW-846

1.0 Scope and Application

This procedure is used to measure the pH of aqueous wastes and multiphasic wastes with at least 20% aqueous phase. This procedure is used in conjunction with SW-846 Chapter 7 to determine the corrosivity characteristic of wastes. An aqueous waste exhibits the characteristic of corrosivity if a representative sample of the waste has a pH $\leq$2 or $\geq$ 12.5 as determined by a pH meter using Method **9040B/C**. This SOP also addresses a corrosivity method modification for soil samples. This modification meets **9040B/C** criteria.

A method detection limit is not appropriate for this method. Results are reported as pH units.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

The pH of the sample is determined electrometrically using a combination electrode. The measuring device is calibrated using a series of standard solution of known pH.

3.0 Definitions

3.1. Corrosivity characteristic – The characteristic that identifies wastes that pose a hazard to human health or the environment due to their ability to:

- mobilize toxic metals if discharged into a landfill environment
- corrode handling, storage, transportation, and management equipment
- destroy human or animal tissue in the event of inadvertent contact.

3.2. Slopes - Change in voltage output resulting from a decade change in the activity of the responding ion. Raw slope values do not provide a good measurement of the electrode's performance due to temperature dependence. The Model 50

reports slope values as temperature referenced to 25 °C. This value reflects the condition of the ion selective electrode (ISE).

3.3. Efficiency Factor - Slope value for electrode divided by its theoretical value. Typical factors for properly functioning electrodes range from 0.90 - 1.05.

3.4. Efficiency - Efficiency factor of the electrode expressed as %. Properly functioning electrode in the Model 50 exhibit efficiencies between 90 - 105%.

3.5. Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.6. Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.7. Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL $<$ LOD $\leq$ LOQ.

3.8. Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.9. Reporting Limit (RL) –

3.9.1. For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.9.2. The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.10. Reporting Units – pH units

3.11. A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.12. SC DHEC – South Carolina Department of Health and Environmental Control

3.13. DoD-QSM – Department of Defense Quality Systems Manual

3.14. Batch – a group of up to 20 field samples of a similar matrix, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate prepared together at the same time.

3.15. LIMS – Laboratory Information Management System

4.0 Interferences

4.1 The corrosivity of concentrated acids and bases, or of concentrated acids and bases mixed with inert substances, cannot be measured. The pH measurement requires some water content.

4.2 The glass electrode, in general, is not subject to solution interferences from color, turbidity, colloidal matter, oxidants, reductants, or moderate (<0.1 M solution) salinity.

4.3 Sodium error at pH levels >10 can be reduced or eliminated by using a low-sodium –error electrode.

4.4 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.5 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation or by calibrating the electrode-instrument system at the temperature of the samples. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. It should, therefore, be noted by reporting both the pH and temperature at the time of analysis.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Fisher Scientific Model 50 pH/ISE meter

6.2 Standard glass- body combination electrode

6.3 Ordinary lab glassware

6.3.1 150 mL plastic specimen cups

6.3.2 50 mL disposable beakers

6.3.3 Magnetic stirrer and stir bars

6.3.4 Analytical Balance

6.4 Element DataSystem® LIMS software by Promium

7.0 Reagents and Standards

Note: Record all reagent and standard preparations on the Corrosivity Determination Work Sheet (See example, Attachment 1) or in LIMS. All purchased chemicals and reagents that do not arrive with an expiration date, must be assigned an expiration date five years from receipt. All lab prepared reagents must be assigned an expiration date one year from preparation.

To obtain information on any standard, access Element, click on "Laboratory," and scroll down and access "Standards." Standards can be sorted by Department. Select the standard you would like to access. The view will show lot number, prepared dates, solvent, vendor, composition, and concentration.

Note: All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise noted. All standards and reagents are subject to change in vendor and in concentration.

7.1 Reagent water-All water used during preparation should be reagent-grade Type I with regard to resistivity of >10 megohm-cm (20th Editions of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Fisher certified (or equivalent) buffers: pH of 2.0, 4.0, 7.0, 10.0 and 12.0.

Note: For corrosivity characterization, the calibration of the pH meter should include a buffer of pH 2 for acidic wastes and a pH 12 buffer for caustic wastes.

7.3 QC reference sample purchased from **NSI** with certificate of analysis.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and SOP 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Samples should be analyzed as soon after receipt as possible.

9.0 Quality Control

9.1 During calibration the value for each of the certified buffer solutions must be within +/- 0.05 pH units.

9.2 Efficiency determined at calibration should be between 90-105%.

9.3 Deviations of more than ± 0.10 pH units in the calibration verification from the true value require recalibration and reanalysis of samples analyzed between the failing buffer and the preceding acceptable buffer.

9.4 Analyze an aqueous QC (laboratory control) sample for each batch of samples. For SC DHEC waters, analyzed the LCS every 10 samples. Obtain prepared solution and acceptance criteria from ERA. Record results and reference numbers in logbook. If acceptance criteria are not met, correct the problem, recalibrate and reanalyze the entire batch.

9.5 Contingency

9.5.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.5.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.5.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.5.4 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 Calibration of the pH meter

10.1.1 The pH meter must be calibrated daily and the calibration recorded on the pH Determination Log (Attachment 1) before sample analysis begins.

10.1.2 Calibrate the meter using the auto recognition standardization method described below and in the Fisher Scientific Model 50 pH Meter Operating Instructions Manual. With this method, the meter automatically corrects the buffer value for the variation of buffer pH with temperature, and it eliminates the need to key and enter a buffer value each time a standardization is performed. Temperature compensation at 25 ^{o}C is provided, independent whether an automatic temperature compensation (ATC) probe is connected. However, if temperature compensation with the ATC probe is not used, all buffers and samples should be at the same temperature.

Note: Multiple readings for each standard is requested in Method**s** **9040B/C**, however, the Fisher Scientific Model 50 pH Meter cannot calibrate using this process. Samples do have multiple readings performed per the method.

10.1.2.1 Use fresh buffer solutions each time the pH meter is calibrated.

10.1.2.2 Stir all buffers and samples with a magnetic stirrer when a measurement is being made.

10.1.2.3 Store the electrode in pH 7.0 buffer when not in use.

10.1.3 From the main screen, press “Standardize”.

10.1.4 Press “2” to clear existing standards.

10.1.5 Press “Standardize” then press “1” to update standards.

10.1.6 Place the electrode into pH 2.0 buffer and turn on the stirrer. Type in the buffer value and press enter. Immerse the electrode in a sufficient volume of solution to cover the sensing elements and allow the electrode to remain in the solution until a stable reading is obtained. Rinse the electrode thoroughly between determinations.

10.1.7 Remove the pH electrode from the pH 2.0 buffer and rinse the electrode thoroughly with DI water. Place the electrode into the pH 4.0 buffer repeating Section 10.1.6.

10.1.8 Repeat Section 10.1.7 for the pH 7.0, 10.0, and 12.0 buffers.

10.1.9 Verify the slope and efficiency after samples have been run.

10.2 Calibration Verification

10.2.1 After the initial calibration, analyze the pH 7.0 buffer and record the value as the LCS Reference St**andar**d on the Corrosivity Worksheet (See Attachment 1).

10.2.2 At a frequency of at least every 10 samples, analyze one of the pH buffer standards that most closely matches the pH range of the previous 10 samples being evaluated. Record the value as a "Buffer Check".

10.3 Calibration of the ATC Probe

10.3.1 On a quarterly basis, the ATC probe is calibrated against the laboratory NIST thermometer following the procedure described in QA SOP 17.12, "Calibrating Thermometers".

11.0 Procedure

11.1 Aqueous Samples

11.1.1 Place the sample or buffer solution in a 50 mL plastic disposable beaker using a sufficient volume to cover the sensing elements of the electrode and to give adequate clearance for the magnetic stir bar.

11.1.2 Thoroughly rinse and gently wipe the electrodes prior to measuring the pH of samples. Note and record the pH and temperature of the sample on the pH Determination worksheet (Attachment 1).

11.1.3 Repeat the measurement on the sample aliquot until values differ by <0.1 pH units. Two or three readings are usually sufficient. Record the additional pH results in the worksheet (Attachment 1).

11.2 Soil Samples

11.2.1 Weigh 20 g of the soil sample in a 150 mL specimen cup, add 20mL of DI H_20, cover and stir the suspension for 5 minutes.

11.2.2 Allow the soil suspension to stand for 1 hour to allow most of the suspended soil to settle out from the suspension.

11.2.3 Immerse the electrode just below the suspension and record the pH and temperature of the sample in the pH Determination Worksheet (Attachment 1).

11.2.4 Repeat the measurement on the sample aliquot until values differ by <0.1 pH units. Two or three readings are usually sufficient. Record the additional pH results in the worksheet (Attachment 1).

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all

sewage discharge permits and regulations is also required. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update **IIB**, 1/**95**, Method 9040B**; Update IIIB, 11/04, Method 9040C**

16.2 Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 1080

16.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.4 Quality Control SOP 13.4, "Numerical Data Reduction"

16.5 Hazardous Waste Management and Safety SOPs: **12.1** "Hazardous Waste Disposal" and **12.2** "Spill Control and Cleanup."

16.6 NELAC Standards, effective July 2003, plus revisions

16.7 Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA-G6, EPA/600/B-07/001, April, 2007.

16.8 CompuChem Quality Manual, Revision **14**, **January 25, 2011**, plus revisions

16.9 Sample Control SOP 4.1, "Receiving Samples"

16.10 Sample Control SOP 4.6, "Storing Samples"

16.11 Quality Assurance SOP 17.12, "Calibrating Thermometers"

16.12 Fisher Scientific Model 50 pH Meter Operating Manual Revision D, 03/92

16.13 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams and Flowcharts

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17.1 Attachment 1 - Example Corrosivity Determination Worksheet

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Attachment 1

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Corrosivity Determination
Method 9040B/C

Worksheet 1 X(7) Rev. 2

SDG: _____
Batch No.: _____

Analyst No.: _____ Date: _____ Time: _____

Standards (pH Units)	pH	Temp (°C)	Slope	Efficiency	Reference Lot No.	Expiration Date
2.00						
4.00						
7.00						
10.00						
12.00			NA	NA		
LCS Reference Stnd.			NA	NA		

*If individual pH readings are not within +/- 0.05 pH units, contact the supervisor. Slope efficiency is 90-105%. Check Std. ±0.05 pH units.
Because pH was not taken in the field, holding time (within 24 hours for 9040B) could not be met.

CompuChem No.	Sample Type	For Soils Only		pH	Temp (°C)	pH	Temp (°C)	Final Result pH	Final Temp (°C)	Comments
		Weight (g)	Water Added (mL)							
								0.00	0.00	
Buffer Check*										
Buffer Check*										

Ref. No. LCS =		Acceptance Criteria = ± 0.05 pH units	TV =	

Reviewed by: _____ Date: _____

pH Meter: Fisher Scientific Accumet® Model 50 **pH MeterElectrode: Orion 8202BN**
Balance: Denver Instr Serial #B034903 **S class weights for Balance calibration:Serial #NCSN050**

* The pH Buffer Check should be representative of the pH range of the previous 10 samples being evaluated.

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

6/28/11 jad

Note: This page subject to change without notice.

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501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 604 SOP Section #: 3.5.14.3 Revision #: 5

SOP Title: Soil and Waste pH by SW-846 Methods 9045C and 9045D

Effective date: (QA fills in) 01-21-2011

- Procedure prepared by: Judith Nozzi Date: 01-21-2011
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 01-21-2011
- Reason for change: Annual review and addition of DoD procedures and protocols
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Edition, Update IIB, 1/95, Method 9045C; SW-846 3RD Edition, Update IIIB, 11/04, Method 9045D; Dept. of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/10

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 604: Soil and Waste pH by SW-846 Methods 9045C and 9045D

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Instrument Procedure 604: Soil and Waste pH by SW-846 Methods 9045C and 9045D

1.0 Scope and Application

Methods 9045C and 9045D are electrometric procedures which have been approved for measuring pH in soils and waste samples. Wastes may be solids, sludges, or non-aqueous liquids. If water is present, it must constitute less than 20% of the total volume of the sample.

A method detection limit study is not required for this analysis.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

The sample is mixed with DI water, and the pH of the resulting aqueous solution is measured.

3.0 Definitions

3.1 Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B). A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL

must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.6 Reporting Units – Standard pH units

3.7 A **Sample Delivery Group (**SDG**)** is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up

to 20 field samples of similar matrices are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.8 Slope - Change in voltage output resulting from a decade change in the activity of the responding ion. Raw slope values do not provide a good measurement of the electrode's performance due to temperature dependence. The Model 50 reports slope values as temperature referenced to 25 ° C. This value reflects the condition of the ion selective electrode (ISE).

3.9 Efficiency Factor - Slope value for electrode divided by its theoretical value. Typical factors for properly functioning electrodes range from 0.90 - 1.05.

3.10 Efficiency - Efficiency factor of the electrode expressed as %. Properly functioning electrode in the Model 50 exhibit efficiencies between 90 - 105%.

3.11 SC DHEC – South Carolina Department of Health and Environmental Control

3.12 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Samples with a very low or very high pH may give incorrect readings on the meter. For samples with a true pH >10, the measured pH may be incorrectly low. Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.2 Temperature fluctuations will cause measurement errors.

4.3 Errors will occur when the electrodes become coated. If an electrode becomes coated with an oily material that will not rinse free, it can be:

- cleaned with an ultrasonic bath
- washed with a detergent followed by several rinses with DI water, then placed in a 1:10 HCl solution and rinsed again
- cleaned according to the manufacturer's instructions

5.0 Safety

Laboratory staff members are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment and Supplies

6.1 Fisher Scientific Model 50 pH/ISE meter

6.2 Standard glass - body combination electrode

6.3 Beaker: 150 mL plastic specimen cups

6.4 Thermometer or temperature sensor for automatic compensation.

6.5 Top loading or analytical balance capable of weighing 0.1 g.

6.6 Magnetic stir plate and stir bars

7.0 Reagents and Standards

7.1 Reagent Water – All water used during preparation must be reagent-grade Type I with regard to resistivity or >10 megohm- cm (19th and 20th Editions of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Fisher certified (or equivalent) buffers: pH of 2.0, 4.0, 7.0, and 10.0

7.3 QC reference samples purchased from ERA with certificates of analyses.

8.0 Sample Preservation and Storage

8.1 Samples must be stored at 4 °C ± 2 °C and should be analyzed as soon as possible.

9.0 Quality Control

9.1 After calibration and at a frequency of at least every 10 samples, analyze the pH 7.0 buffer as calibration verification. Deviations of more than ± 0.10 pH units from the true value require recalibration and reanalysis of samples analyzed between the failing buffer and the preceding acceptable buffer.

9.2 Analyze an aqueous blank spike for each batch of samples. Obtain prepared solution and acceptance criteria from ERA. Record the results and reference

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numbers in the pH Determination **Work Sheet** (Attachment 1). If acceptance criteria are not met, correct the problem, recalibrate and reanalyze the entire batch.

9.3 A duplicate sample must be analyzed with every 10 samples. Deviations of more than ± 0.10 pH units between duplicates require recalibration and reanalysis of all associated samples.

9.4 Efficiency determined at calibration must be between 90 - 105%.

9.5 Contingency

9.5.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.5.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.5.3 Any other issues that potentially effect data quality must be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 The pH meter must be calibrated and recorded on the pH Determination **Work Sheet** before this analysis begins.

10.2 The value for each of the certified buffer solutions must be within ± 0.05 pH units. Use fresh buffer solutions each time the pH meter is calibrated.

10.3 Calibrate the meter using either the auto recognition standardization method described below and in the Fisher Scientific Model 50 pH Meter Operating Instructions Manual. With this method, the meter automatically corrects the buffer value for the variation of buffer pH with temperature and it eliminates the need to key and enter a buffer value each time standardization is performed. Temperature compensation at 25° C is provided. The automatic temperature compensation (ATC) probe must be connected.

Stir all buffers and samples with a magnetic stirrer while a measurement is being made.

10.3.1 From the main screen, press "Standardize".

10.3.2 Press "2" to clear existing standards.

10.3.3 Press "Standardize" then press "1" to update standards.

10.3.4 Place the electrode into pH 2.0 buffer and turn on the stirrer. Type in the buffer value and press enter. Immerse the electrode in a sufficient volume of solution to cover the sensing elements and allow the electrode to remain in the solution until a stable reading is obtained. Rinse the electrode thoroughly between determinations.

10.3.5 Repeat steps in 10.3.4 using buffer solutions at pH 4.0, 7.0 and 10.0 buffers.

10.4 Store the electrode in pH 7.0 buffer when not in use.

10.5 Check the slope and efficiency. Efficiency must be 90 - 105%.

11.0 Procedure

11.1 Weigh 20 g of the soil or waste sample in a 150 mL specimen cup, add 20 mL of DI H_20, cover and stir the suspension for 5 minutes. Additional dilutions are allowed when working with hygroscopic soil and salts or other problematic matrices. All sample volumes must be documented on the **pH Determination Work Sheet**. (See **Attachment** 1.)

11.1.1 Soil samples must be allowed to stand for about 1 hour to allow the suspended waste to settle out from the suspension or filtered or centrifuged off the aqueous phase for pH measurement. The option used must be documented on the **work sheet**. If the option to allow the samples to settle is used, the time must also be documented on the **work sheet**.

11.1.2 Waste samples must be allowed to stand for about 15 minutes to allow the suspended waste to settle out from the suspension or filtered or centrifuged off the aqueous phase for pH measurement. The option used must be documented on the **work sheet**. If the option to allow the samples to settle is used, the time must also be documented on the **work sheet**.

11.1.2.1 If the waste is hygroscopic and absorbs all the DI water, begin the procedure again, using 20 g of waste and 40 mL of DI water.

11.1.2.2 If the liquid layer is multiphasic, decant the oily phase and measure the pH of the water phase.

11.3 Immerse the electrode just below the suspension and record the pH and temperature of the sample in the pH Determination **Work Sheet** (Attachment 1).

11.4 Thoroughly rinse the electrode between samples.

12.0 Data Analysis and Calculations

Calculations must be consistent with the QC SOP **13.4** : **"**Numerical Data Reduction**"**.

12.1 Report the soil or waste sample results as "soil or waste pH measured in water at ____ ^{o}C, where "____ ^{o}C" is the temperature at which the test was conducted.

13.0 Method Performance

This method was validated through in-house laboratory studies of precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, H_2SO_4, NaOH, or Zn Acetate are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update IIB, 1/95, Method 9045C and Update IIIB, 11/04, Method 9045D

16.2 QC SOP **13.6**: **"**Proper Documentation Procedures**"**

16.3 QC SOP **13.4**: **"**Numerical Data Reduction**"**

16.4 "Less is Better: Laboratory Chemical Management for Waste Reduction," American Chemical Society Department of Government Relations and Science Policy, 1155 16th Street, N.W., Washington DC, 20036, (202) 872-4477.

16.5 Hazardous Waste Management **and** Safety SOPs **12.1**: "Hazardous Waste Disposal" and **12.2** "Spill Control **and** Cleanup."

16.6 NELAC Standards, effective July 2003, plus revisions

16.7 **Guidance for Preparing Standard Operating Procedures (SOPs), EPA QA-G6, EPA/600/B-07/001, April, 2007**.

16.8 CompuChem Quality Manual, Revision **13**, **November 17, 2009**, plus revisions

16.9 Sample Control SOP 4.1, "Receiving Samples"

16.10 Sample Control SOP 4.6, "Storing Samples"

16.12 Fisher Scientific Model 50 pH Meter Operating Manual Revision D, 03/92

16.13 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams, **and** Flowcharts

17.1 Attachment 1 – **Sample** pH Determination **Work Sheet**

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Attachment 1

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Worksheet 1 HH Rev. 1

pH Determination
SM4500-H B or 9040B (water)
9045C/D (soil)
(Circle which method was used.)

Date: _____ /Time: _____

Batch No.: _____

SDG: _____

Analyst No.: _____

Standards (pH Units)	pH	Temp (°C)	Slope / Efficiency	Reference Lot No.	Expiration Date	Comments
2.00						Efficiency (90-105%)
4.00						Efficiency (90-105%)
7.00						Efficiency (90-105%)
10.00						Efficiency (90-105%)
12.00			NA			
LCS Reference Stnd.		NA	NA			Acceptance Criteria ± 0.05 pH Units

		FOR SOILS ONLY				
CompuChem No.	Sample Type	Weight (g)	Water Added (mL)	pH Result	Temp (°C)	Comments
Buffer Check*						
Buffer Check*						

* The pH Buffer Check should be representative of the pH range of the previous 10 samples being evaluated.

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Reviewed by: _____ Date: _____

pH Meter: Fisher Scientific Accumet® Model 50
Balance: Denver Instr Serial #B034903

pH MeterElectrode: Orion 8202BN
S class weights for Balance calibration:Serial #NCSN050

10/17/10:jad

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: SPP-1050 SOP Section #: 3.5.18.1 Revision #: 8

SOP Title: Reactive (Total Releasable) Sulfide by SW-846 Chapter 7, Section 7.3.4 and Method 9034

Effective date: (QA fills in) 11-29-2010

- Procedure prepared by: _____ Date: 11-29-2010
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Meiner Date: 11-29-2010
- Reason for change: Addition of DoD protocols and annual Review
- This procedure meets the requirements of the following approved method references: SW-846, 3rd Edition, Update III, 12/96, Chapter 7 and Method 9034; DoD Quality Systems Manual for Environmental Laboratories, Ver ~~4.1~~ 4.2 ~~4/22/08~~ 10/26/10

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Preparation Procedure -1050: Reactive (Total Releasable) Sulfide by SW-846 Chapter 7, Section 7.3.4 and Method 9034

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Sample Preparation Procedure -1050: Reactive (Total Releasable) Sulfide by SW-846 Chapter 7, Section 7.3.4 and Method 9034

1.0 Scope and Application

1.1 This Standard Operation Procedure (SOP) describes the preparation and determination of the total release of hydrogen sulfide in wastes upon contact with an aqueous acid. Samples are prepared using the SW-846 procedure in Chapter 7, section 7.3.4 and the sulfide quantified using the titration procedure in Method 9034. This procedure is applicable to all wastes, with the condition that wastes that are combined with acids do not form explosive mixtures. The current EPA guidance level for total releasable sulfide is 500 mg H_2S/kg waste.

1.2 This procedure releases only the evolved hydrogen sulfide at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

1.3 The reporting limit is set at one-fourth the regulatory level or 125 mg/Kg. Only values above this limit are reported.

1.4 Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

1.5 This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

An aliquot of acid is added to a fixed weight of waste in a closed system. The generated gas is swept into a scrubber. The analyte is quantitated by titration

3.0 Definitions

3.1 Reactivity characteristic - According to the regulation in 40 CFR 261.23, a solid waste exhibits the characteristic of reactivity if a representative sample of the waste has any of the properties listed below. This definition is intended to identify wastes that, because of their extreme instability and tendency to react

violently or explode, pose a problem at all stages of the waste management process.

- It is normally unstable and readily undergoes violent change without detonating.
- It reacts violently with water.
- It forms potentially explosive mixtures with water.
- When mixed with water, it generates toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
- It is a cyanide- or sulfide-bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes in a quantity sufficient to present a danger to human health or the environment.
- It is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement.
- It is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure.
- It is a forbidden explosive, as defined in 49 CFR 173.51, or a Class A explosive, as defined in 49 CFR 173.53, or a Class B explosive, as defined in 49 CFR 173.88

3.2 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined **from** analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.) **A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.3 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.4 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.5 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.6 Reporting Limit (RL) –

3.6.1 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.7 Reporting Units – mg/Kg or mg/L

3.8 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for DoD-QSM and 10% for SC DHEC. If samples are batched together from different sites, project-specific QC must be processed.

3.9 SC DHEC – South Carolina Department of Health and Environmental Control

3.10 DoD-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 The iodometric method suffers interferences from reducing substances that react with iodine, including thiosulfate, sulfite, and various organic compounds.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and MSDS are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 Apparatus for Reactive Sulfide (Attachment 1)

6.2 500 **mL** beakers

6.3 Magnetic stirrer

6.4 Stir bars

6.4 Pipettes

6.5 Volumetric flasks, various sizes

6.6 Pyrex Class A burets, 50 **mL**

7.0 Reagents and Standards

Preparation of all reagents and standards is documented in the Reagent/Standard/QC Preparation Logbook (Attachment 3).

Note: **All reagents and standards must be ACS (American Chemical Society) grade or equivalent.** All standards and reagents are subject to change in vendor and in concentration. **The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.**

7.1 Reagent water - All water used during preparation should be reagent-grade Type I with regard to resistivity of >10 megohm (20th Editions of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

7.2 Sulfuric acid **H_2SO_4, Concentrated**

7.2.1 **Sulfuric acid (0.1N) -** Add 2.8 **mL** concentrated H_2SO_4 to reagent water and dilute to 1 L.

7.2.2 Sulfuric Acid (0.01N) **- Add 100 mL of 0.1N H_2SO_4 to reagent water and dilute to 1 L.**

7.3 Hydrochloric acid HCl**, Concentrated**

7.3.1 **Hydrochloric acid (6N) HCl –** Add 50**0 mL** concentrated HCl to reagent water and dilute to 1 L.

7.4 Sulfide reference solution (1000 mg/L) **- Purchased commercially prepared from NSI.**

7.5 Sodium hydroxide**, Pellets**

7.5.1 **Sodium hydroxide solution (0.25N) -** Dissolve 10 g of NaOH pellets in reagent water and dilute to 1 L.

7.6 Iodine solution (0.025N) **- Purchased commercially from VWR.**

7.7 Sodium Thiosulfate (0.25N) **- Purchased commercially from VWR.**

7.3 Starch indicator **- Purchased commercially from VWR.**

8.0 <u>Sample Preservation and Storage</u>

8.1 The sample must be stored in the dark, refrigerated at 4 °C ± 2 °C and run as soon as possible but within 7 days of sampling. The holding time is 7 days from date of collection.

8.3 Samples are preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

9.0 Quality Control

9.1 Method Blank (Preparation or Reagent Blank): The blank is used to establish that the reagents are free from contamination. One method blank must be analyzed with every preparation batch of up to twenty samples. The method blank must be identified on the run log.

9.1.1 If any value present in the method blank exceeds the reporting limit, the entire batch must be reprocessed using clean glassware.

9.1.2 For the **DoD**-QSM the preparation blank must not contain sulfide at a level above one half the reporting limit.

9.2 Laboratory Control Sample (LCS): The LCS solution is 1.0 mL of the sulfide reference solution **in 10 mL reagent water**. The LCS must be prepared with every batch of up to twenty samples. Accuracy of the process is determined from the LCS recovery.

9.2.1 The result of the LCS must agree within 50 - 175% of the true value. The true value is 1000 mg/Kg. When the LCS is outside this range, the system must be checked, the source of error identified and corrected, a new LCS prepared, and the associated batch of samples must be reprocessed.

9.3 A matrix spike is prepared for every batch of up to 20 samples. Prepare the matrix spike using **1.0** mL of sulfide reference solution and 10 g of sample. The spike recovery should be within 50 – 175%. If the recovery of the matrix spike is not within limits but the LCS meets acceptance criteria, reanalysis of is not necessary.

9.4 Contingency

9.4.1 If due to a lab accident or to QC failures, a re-preparation and analysis are required for the sample and insufficient sample volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.4.2 If persistent contamination occurs in the laboratory, analysis must be halted until the source of the contamination can be identified and isolated. When the contamination issue is resolved, samples analysis may proceed.

9.4.3 Any other issues that potentially effect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization

N/A

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures".

Record all data in the Reactive Sulfide Preparation and Analysis Log (Attachment 2). Include all pertinent information such as sample size, date of analysis, and sample ID, etc. As the analysis proceeds, discrepancies or deviations must be written in the logbook immediately.

11.1 Prepare the sample distillate

11.1.1 S**et** up apparatus. (See example in Attachment 1)

11.1.2 Add 50 **mL** of 0.25N NaOH solution to a calibrated scrubber.

11.1.3 Close the system and adjust the flow of nitrogen gas, using the rotometer. The flow rate should be 60 **mL**/min.

11.1.4 Add 10 g of the waste to be tested to a 500 mL round bottom flask.

11.1.5 Seal all ground glass fittings with Teflon® tape or Nalgene® clamps.

11.1.6 With the nitrogen flowing, add 250 **mL** of (0.01N) H_2SO_4 into the flask, while starting the 30 minute test period.

11.1.7 Begin stirring while the acid is entering the round bottom flask. The stirring should remain at a constant rate throughout the test and should not create a vortex.

11.1.8 After 30 minutes, close off the nitrogen and disconnect the scrubber.

11.1.9 Document the 30 minute Reactive Time on the preparation and analysis log (Attachment 2).

11.2 Titrate the distillate.

11.2.1 Measure 20 **mL** of scrubber solution into a 500 **mL** beaker. Bring to 200 **mL** with reagent water.

11.2.2 Add a known amount of 0.025N iodine solution **(I_2)** in excess of the amount of sulfide present.

11.2.3 Add 2 **mL** 6N HCl and approximately 1 **mL** of starch indicator until dark blue.

11.2.4 Titrate the solution in the beaker with 0.025N sodium thiosulfate solution until the blue color disappears. Record the volume of titrant on the analysis log.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculate the concentration of sulfide in the scrubber using the following equation:

Sulfide (mg.L) =

$$\frac{(mL\ I_2\ x\ 0.025N\ I_2) - (mL\ titrant\ x\ 0.025N\ titrant)\ x \left(\frac{32.06\ g}{2\ eq.}\right)}{scrubber\ volume\ (L)}$$

12.2 Calculate the specific rate of release of H_2S using the following equation:

$$R = \text{Specific rate of release (mg/Kg/sec)} = \frac{A\ x\ L}{W\ x\ S}$$

where: A = Concentration of H_2S in scrubber (mg/L) (from **Section** 12.1)

L = Volume of solution in scrubber (L) (The formula used in the spreadsheet software accommodates the 20 **mL** volume used for titration from the 50 **mL** scrubber solution.)

W = Weight of waste used (kg)

S = Time of experiment (30 minutes) in seconds (time N_2 stopped - time N_2 started)

12.3 Calculate the total releasable H_2S (mg/kg) using this equation:

Total releasable H_2S (mg/kg) = R x S

where: R = Specific rate of release (mg/kg/sec) from **Section** 12.2

S = Time of experiment (30 minutes) in seconds

12.4 Relative Percent Difference (RPD):

$$\text{RPD} = \frac{|S - D|}{(S + D)/2} x\,100$$

where: S = concentration in original sample
D = concentration in duplicate sample

12.5 Data Review and Verification

12.5.1 Initial review is performed by the analyst who also generates the final report. A peer or supervisory review is performed prior to release of analytical results to the client.

13.0 <u>Method Performance</u>

This method was validated through in-house laboratory studies of precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, "Hazardous Waste Disposal" for procedures on pollution prevention.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Third Edition, Chapter 7, Update III, and Method 9034, 12/96.

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 NELAC Standards, effective July 2003, plus revisions

16.4 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.5 Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples"

16.6 CompuChem Quality Manual, Revision **13**, **November 17, 2010**, plus revisions

16.7 **Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010**

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1- Apparatus **diagram** used for this procedure

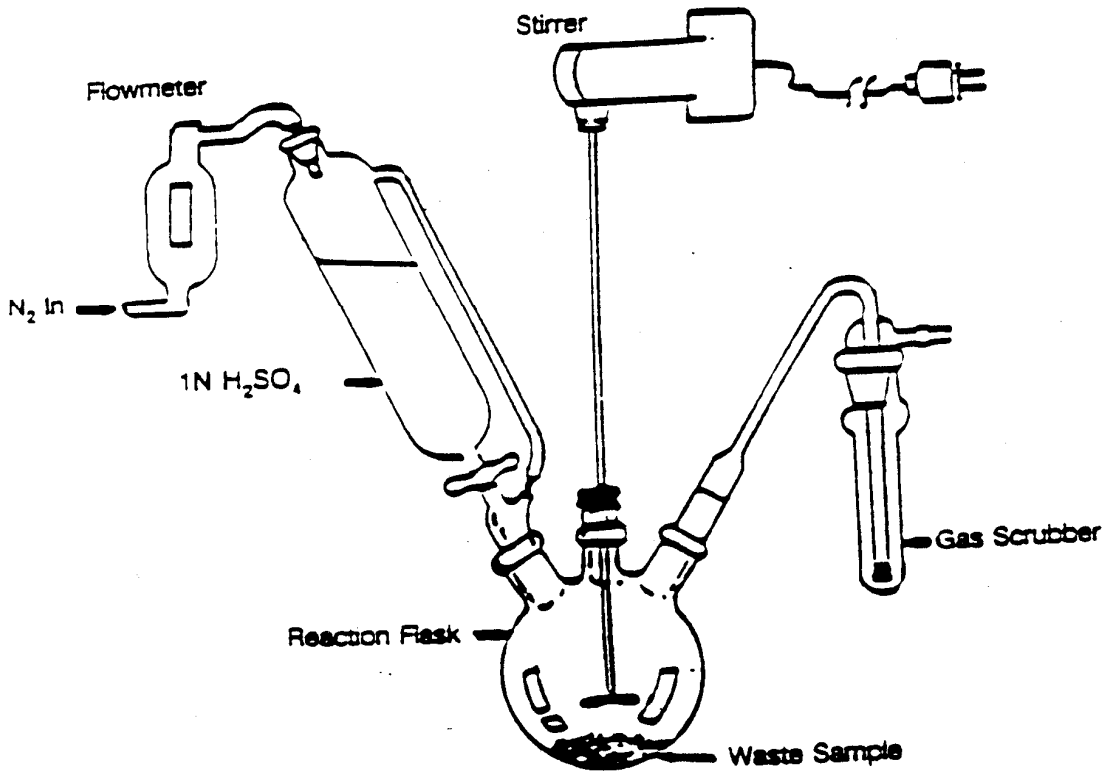
17.2 Attachment 2- **Example** Reactive Sulfide Preparation and Analysis Log

17.3 Attachment 3 – **Example** Reagent/QC Preparation Log

Attachment 1

APPARATUS TO DETERMINE HYDROGEN SULFIDE RELEASED FROM WASTES

Attachment 1



Attachment 2

CompuChem
A Division Of Liberty Analytical Corp.

Reactive Sulfide Preparation and Analysis
Method SW-846 Chapter 7/9034

Date: _____/Time: _____

Worksheet 1 W(5) R1

SDG: _____

Analyst No.: _____

Batch No.: _____

CompuChem No.	Client ID	Sample Type	A Initial Sample (kg)	B Total Scrubber Vol. (L)	C Scrubber Vol. Titrated (mL)	D Iodine Added (mL)	E Titrant Vol. (mL)	Results Total Releasable H_2S in water/soil (mg/kg)	Comments
			0.01	0.05	20			0.00	

Reactive Time	LCS Reference	MS Reference
Start: _____	LCS: Add 1 mL of 10,000 mg/L Sulfide solution into 10 mL of reagent water	MS: Add 1 mL of the 10,000 mg/L Sulfide solution into 10 g of sample.
Finish: _____	See Worksheet 1 M(8) Rev.3 for LCS/MS Ref. No., Manufacturer Lot No., and Exp. Date Sequence File Name: _____	

Duplicates ± 20%	%RPD=		LCS Range 50-175	% Rec =	

Total Releasable H_2S, mg/kg = (D - E) x $\frac{0.025N}{C}$ x $\frac{32.06\ g}{2\ eq.}$ x $\frac{1000\ mg}{1\ g}$ x $\frac{B}{A}$ = (D - E) x 100.1875*

*when B=0.05 & C=20

Balance ID:_____ Reviewed By: _____ Date:_____

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

11/24/10:jad

Note: This page subject to change without notice.

Attachment 3

Reagent/QC Preparation for Reactive Sulfide Analysis Seq. File Name:_____

COMPUCHEM a division of Liberty Analytical Corp. WORKSHEET 1 M(8) Rev. 3

REAGENTS*	REFERENCE NUMBER	Manufacturer	Lot Number	Expiration Date
Sulfuric Acid (H_2SO_4) - Concentrated				
Sodium Hydroxide (NaOH)				
Starch Indicator				
Iodine Solution				
Hydrochloric Acid (HCl) - Concentrated				
Sodium Thiosulfate				
Sulfide LCS/MS				

REAGENT/STANDARD LOT NUMBER**	PREPARATION INSTRUCTIONS
0.1N H_2SO_4-	Add 2.8 mL concentrated H_2SO_4 to reagent water and dilute to 1 L.
0.01N H_2SO_4-	Add 100 mL of 0.1N H_2SO_4 to reagent water and dilute to1 L.
6N HCl-	Add 500 mL concentrated HCl to reagent water and dilute to 1 L.
0.25N NaOH-	Dissolve 10 g of NaOH pellets in reagent water and dilute to 1 liter.

Reagent/Standard Solutions Prepared By: _____ Date: _____

Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

The presence of the Chemist's/Analyst's employee ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log **Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

Page 1 of 1

11/24/10:rem/v

Note: This page subject to change without notice.

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501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 610 SOP Section #: 3.5.19.1 Revision #: 8

SOP Title: Non-filterable Residue, Total Suspended Solids, by Standard Methods for the Examination of Water and Wastewater

Effective date: (QA fills in) 07-28-2010

- Procedure prepared by: _____ Date: 072810
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Nieva Date: 07-28-2010
- Reason for change: annual Review, add DoD-QSM Procedures and Protocols
- This procedure meets the requirements of the following approved method references: Standard Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 2540D; DoD Quality Systems Manual for Environmental Laboratories, Version 4.1, 4/22/2009

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 610: Non-filterable Residue, Total Suspended Solids, by Standard Methods for the Examination of Water and Wastewater

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Instrument Procedure 610: Non-filterable Residue, Total Suspended Solids, by Standard Methods for the Examination of Water and Wastewater

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the determination total suspended solids in drinking, surface, and saline waters, and domestic and industrial wastes waters **by Standard Methods for Examination of Water and Wastewater, Method 2540D.**

A method detection limit is not applicable to this determination. The reporting limit is 2.5 mg/L and is adjusted based on the volume of sample used. This is discussed in detail below.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A well-mixed sample is filtered through a glass fiber filter disc (47 mm) and the non-filterable residue on the filter is dried to a constant weight at 103 – 105 °C.

3.0 Definitions

3.1 Non-filterable residue - the solid mass retained by a glass fiber filter disc and dried to a constant weight at 103 – 105 °C.

3.2 Reporting Units – mg/L

3.3 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or

- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.4 SC DHEC – South Carolina Department of Health and Environmental Control

3.5 **DoD**-QSM- Department of Defense Quality Systems Manual

3.6 Constant weight – Weight obtained when weight loss is < 4% difference or < 0.5 mg (0.0005 g), whichever is less.

3.7 **Method Detection Limit (MDL) - The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from the analysis of a sample in a given matrix containing the analyte. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present. Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater that zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B.)**

3.8 **Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.9 **Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM,**

non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.10 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.11 Reporting Limit (RL) –

3.11.1 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

4.0 Interferences

4.1 Filtration apparatus, filter material, and washing and drying temperatures are specified because these variables have been shown to affect the results.

4.2 Samples high in dissolved solids, such as saline waters, brines, and some wastes, may be subject to a positive interference. Care must be taken in selecting the filtering apparatus so that washing the filter and any dissolved solids in the filter minimizes this potential interference.

4.3 Exclude large floating particles/submerged non-homogeneous materials, if not representative of the sample.

5.0 Safety

5.1 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

5.2 Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and the MSDS are located in the Quality Assurance department.

6.0 Equipment and Supplies

6.1 "ProWeight Filters" pre-weighed 47 mm glass fiber filters catalog number F93447mm from Environmental Express, or equivalent

6.2 Suction flasks

6.3 Drying oven **-** 103 **–** 105 °C

6.4 Dessicator, provided with a desiccant containing a color indicator of moisture concentration

6.5 Analytical balance, capable of weighing to 0.1 mg

6.6 Aluminum weighing dishes

6.7 Magnetic stirrer with TFE stirring bar

6.8 Wide-bore pipettes

6.9 Graduated cylinders

7.0 Reagents and Standards

7.1 Reagent water - All water used during preparation should be reagent-grade Type I with regard to resistivity of >10 megohm-cm (20th Edition of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

7.2 LCS - Mid level solids standard obtained from NSI.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Preservation of the sample is not practical; analysis should begin as soon as possible.

- **Refrigerate** at 4 °C (± 2 °C), to minimize microbiological decomposition of solids, is recommended. The holding time is seven days.

8.3 Shake thoroughly to achieve homogeneity.

9.0 Quality Control

9.1 Analyze a method blank for every batch of up to 20 samples. For the SC DHEC, the method blank is prepared every 10 samples. The blank must measure less than 1 mg/L of non-filterable residue.

9.1.1 If the blank exceeds 1 mg/L, the entire sample batch associated with the blank must be re-prepared and reanalyzed.

9.2 At least one duplicate sample analysis should be performed for every 10 samples or for each SDG, whichever is more frequent. The sample and duplicate should agree within 5% of their average weight.

9.2.1 If the sample and the duplicate do not agree within 5% of their average weight, the data may be reported and should be qualified.

9.3 A laboratory control sample (LCS) with every batch of up to 20 samples. For the SC DHEC the LCS is prepared every 10 samples. The LCS is commercially available. Acceptable limits are 90 - 110% of the true value.

9.3.1 If the LCS recovery falls outside acceptance limits, the entire batch must be evaluated and repeated as needed.

9.4 The balance calibration is checked at the end of each weighing sequence. This is done by weighing a class S weight and recording the result in the logbook (**Attachment** 1).

9.8 Contingency

9.8.1 If due to a lab accident or to QC failures a re-preparation and re-analysis are required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for directions on how to proceed.

9.8.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analysis may proceed.

9.8.3 Any other issues that potentially affect data quality should also be addressed with the Project Manager.

10.0 Calibration and Standardization

10.1 Ensure the balance has been calibrated for the day prior to use, refer to Quality Control SOP 13.17, "Analytical Balance Calibration and Maintenance".

10.2 The balance, oven, and weight set serial numbers are recorded in the log for reference.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP, "Proper Documentation Procedures". All fields must be completed in the Residue Determination Log (Attachment 1.)

11.1 Preparation of glass fiber filter disc

11.1.1 Filters arrive in the laboratory pre-weighed, and dried. An initial weight is located on the tin. Remove the tin and weigh the filter. Record the weight on the Residue Determination Log.

Note: The filter weight does not include the tin.

11.2 Selection of sample volume

11.2.1 For a 47 mm diameter filter, filter 100 mL of sample. If the weight of captured residue is less than 1.0 mg, the sample volume must be increased to provide at least 2.5 mg of residue. Minimum field sample volume is 300 mL up to 1000 mL. Additional aliquots of sample may be added to the filter when increasing the sample amount to collect more residue.

Note: If other filter diameters are used, start with a volume of sample equal to 7mL/cm^2 of filter area and collect at least 2.5 mg.

11.2.2 If greater than 200 mg are contained on the filter, a lesser sample volume must be used that results in a filter charged with 1-200 mg sample residue.

11.2.3 The reporting limit is adjusted based on the sample volume used. If there is not enough sample volume provided to filter 1000 mL the reporting limit should be adjusted accordingly, and the value obtained is a qualified estimated value. The reporting limit for each sample is based on the volume of sample filtered. For example, a reporting limit of < 10.0 mg/L would be reported if only 250 mL of sample was available for filtering. If one liter is filtered and the residue collected is < 2.5 mg, the reporting limit is < 2.5 mg/L.

11.2.4 The true value may vary with each lot of LCS solution purchased from the vendor. The volume of LCS solution filtered depends on the true value and must always yield > 2.5 mg of residue charged to the filter.

11.3 TSS determination

11.3.1 Using forceps, remove filter from tin and assemble the filtering apparatus and begin suction. Wet the filter with a small volume of reagent water to seat it against the fritted support.

11.3.2 While thoroughly mixing the sample (using a magnetic stirrer and TFE stirring bar), pipet away from the vortex, a 100 mL sample aliquot into a graduated cylinder. Pour the sample aliquot into the filter.

Note: As noted above this volume may need to be adjusted and the initial volume can be varied depending on the appearance of the sample, i.e., if suspended solids are observed, turbidity, cloudiness, etc. decrease the volume. Continue to add additional sample aliquots to the filter until at least 1.0 mg residue is collected.

11.3.3 Filter one liter of reagent water for the method blank.

11.3.4 Filter 100 - 200 mL of the LCS solution. This volume may vary with each lot as discussed above.

11.3.5 Filter a sample duplicate.

11.3.6 With the suction on, wash the graduated cylinder, filter, non-filterable residue, and filter funnel wall with three 10 mL portions of reagent water allowing complete drainage between washings.

11.3.7 Remove all traces of water by continuing to apply the vacuum for about 3 minutes after the water has passed through.

Note: The total volume of the wash water used should equal approximately 2mL/cm^2. For a 47 mm filter the total volume is 30 mL.

11.3.8 If the filtration rates drop rapidly or if the filtration times exceeds 5 - 10 minutes, increase the filter diameter or decrease the sample volume.

11.3.9 Carefully remove the filter from the support and transfer it to an aluminum weighing dish. Dry at least one hour at 103 – 105 ^{o}C. Cool in a dessicator and weigh. Repeat the drying cycle until a constant weight is obtained (< 4% difference or < 0.5mg, i.e., 0.0005 g, between successive weighings.) The drying procedure includes re-heating, re-dessicating and re-weighing.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Concentration for non-filterable residue

$$Non-\ filterable\ residue, mg/L = \frac{(A-B)*1000}{C}$$

Where: A = weight of filter (or filter and crucible) + residue in mg
B = weight of filter (or filter and crucible) in mg
C = mL of sample filter

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible,

laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Standard Methods the Examination of Water and Wastewater, 20th Edition (1998), Method 2540D

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Edition, Method 1080

16.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.4 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 NELAC Standards, **effective July** 2003**, plus revisions**

16.7 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995

16.8 CompuChem Quality Manual, Revision **13**, **November 17, 2009, plus revisions**

16.9 Quality Control SOP 13.17, "Analytical Balance Calibration and Maintenance"

16.10 Department of Defense Quality Systems Manual for Environmental Laboratories, Version **4.1**, **April 22, 2009**

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example** Residue Determination Log

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Attachment 1

CompuChem
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Non-Filterable Residue (TSS) SM 2540D

Worksheek No. 1 E(8) Rev. 2

Analyst No.: _____ Date/Time: _____ SDG #: _____

CompuChem ID	Client ID	Sample Type	Filter ID Number	Initial Filter Weighing- First (g)	Sample Volume (mL)	Final Filter Weighing- First (g)	Final Filter Weighing Second (g)	Final Filter Weighing- Third (g)	Net Residue (Final weight - dish) (g)	Results (net residue / sample vol) (mg/L)	Comment
				1.0000	100	2.0000	NA	NA	1.0000	10000.00	

Balance Calibration (200.0000 g)	
Initial Weighing	g
Final Weighing	g
Tolerance +/- 0.2000 g	

Shading has been applied to unused portions of this worksheet as a substitute for 'Z-out.

Record of Tempature/Time	
Date samples in oven:	
TIme samples in oven:	
Tempature of oven In:	
Date samples out of oven:	
TIme samples out of oven:	
Tempature of oven out:	

LCS Reference
LCS: Mid Level Solids Standard
REF #

Reviewed BY: _____ Date: _____

Filter Vendor: _____ Filter Lot: _____

Constant Weight is obtained when the weight loss of successive weighting is less than 4 percent or less than 0.5 mg, whichever is less.

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Balance used for weights: Denver Instr Serial #B034903 S class weights for balance calibration:Serial #NCSN050 Fisher Oven Serial #007C 9/14/10:jad

Note: This page subject to change without notice.

SOP 3.5.19.2

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Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) 9/3/10

- Procedure Code: VP609 SOP Section #: 3.5.19.2 Revision #: 10

SOP Title: Filterable Residue, Total Dissolved Solids by Standard Method for the Examination of Water and Wastewater

Effective date: (QA fills in) 09/08/10

- Procedure prepared by: _____ Date: 09/08/10
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob McGiver Date: 09-04-2010
- Reason for change: annual review and addition of DoD protocols
- This procedure meets the requirements of the following approved method references: Standard Method for the Examination of Water and Wastewater, 20th Edition (1998), Method 2540C; DoD Quality Systems Manual for Environmental Laboratories, Version 4.1, 4/22/2009

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 609: Filterable Residue, Total Dissolved Solids, by Standard Method for the Examination of Water and Wastewater

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Instrument Procedure 609: Filterable Residue, Total Dissolved Solids, by Standard Method for the Examination of Water and Wastewater

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the determination of filterable residue in drinking, surface, and saline waters. The practical range of the determination is 10 to 20,000 mg/L.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary of Method

A well-mixed sample is filtered through a glass fiber filter. The filtrate is evaporated and dried to a constant weight at 180 °C.

3.0 Definitions

3.1 Filterable Residue - solids that will pass through a glass fiber filter and are then dried to a constant weight at 180 °C.

3.2 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.3 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL

must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.4 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.5 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.6 Reporting Limit (RL) –

3.6.1 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.7 Reporting Units – mg/L

3.8 A Sample Delivery Group (SDG) is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The **DoD**-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together. If samples are batched together from different sites, project-specific QC must be processed.

3.9 SC DHEC – South Carolina Department of Health and Environmental Control

3.10 **DoD**-QSM – Department of Defense Quality Systems Manual

4.0 Interferences

4.1 Mineralized water containing large concentrations of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and will require longer drying and desiccation time and rapid weighing.

4.2 Samples containing high concentrations of bicarbonate will require careful, and possibly prolonged, drying at 180° C to ensure that all bicarbonate is converted to carbonate.

4.3 Too much residue in the evaporating dish will crust over and entrap water that will not be driven off during drying. Total residue should be limited to about 200 mg.

4.4 Exclude large floating particles/suspended non-homogeneous material, if not representative of the sample.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. During sample preparation safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets (MSDS) for reagents used in the laboratory. The Chemical Hygiene Plan and the MSDS are located in the quality assurance department.

6.0 Equipment and Supplies

6.1 Glass fiber filter discs - 4.7 cm or 2.1 cm, without organic binder

6.2 Filter holder -membrane filter funnel or Gooch crucible adapter

6.3 Suction flask - 500 mL

6.4 Gooch crucibles - 25 mL (if 2.1 cm is used)

6.5 Evaporating dishes - porcelain, 100 mL volume (Vycor or platinum dishes may be substituted)

6.6 Drying oven – 180 °C ± 2 °C

6.7 Desiccator, provided with desiccant contain color indicator of moisture concentration

6.8 Analytical balance - capable of weighing to 0.1 mg.

6.9 Muffle furnace for operation at 550 ºC

6.10 Magnetic stirrer with TFE stirring bar

6.11 Wide-bore pipettes

6.12 Graduated cylinder

7.0 Reagents and Standards

7.1 Reagent water - All water used during preparation should be reagent-grade Type I with regard to resistivity of >10 megohm-cm (20th Edition of Standard Methods, Method 1080), and referred throughout this SOP as reagent water.

8.0 Sample Preservation and Storage

8.1 Samples are preserved and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Analysis of the sample should begin as soon as possible since sample preservation is not practical. Refrigerate the samples at 4° C to reduce microbiological decomposition. The holding time is 7 days.

9.0 Quality Control

9.1 A method blank is analyzed with every batch of up to 20 samples. SC DHEC requires a method blank with every 10 samples.

9.1.1 The method blank must measure less than 10 mg/L of filterable residue. If the method blank exceeds 10 mg/L, the entire sample batch associated with the blank must be re-prepared and reanalyzed.

9.2 For every 10 samples, at least one duplicate sample analysis should be performed. The sample and duplicate should agree within 5% of the average weight.

9.2.1 If the sample and the duplicate do not agree within 5% of the average weight, a Quality Assurance (QA) notice is required.

9.3 A laboratory control sample (LCS) is analyzed with every batch of up to 20 samples. SC DHEC requires an LCS every 10 samples. The LCS is purchased with a certificate of traceability to NIST.

9.3.1 Acceptance limits are 90 - 110% of the true value. If the LCS recovery falls outside acceptance limits, the entire batch must be evaluated and repeated as needed.

9.4 The balance calibration is checked at the end of each weighing sequence. This is done by weighing a class 1 weight and recording the result in the logbook (Attachment 1).

9.5 Contingency

9.5.1 If due to a lab accident or to QC failures a re-preparation and re-analysis are required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for directions on how to proceed.

9.5.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analysis may proceed.

9.5.4 Any other issues that potentially affect data quality should also be addressed with the Project Manager.

10.0 <u>Calibration and Standardization</u>

10.1 Ensure the analytical balance has been calibration for the day prior to use. Refer to Quality Control SOP 13.17, “Analytical Balance Calibration and Maintenance.”

10.2 The balance, oven, and weight set serial numbers are recorded in the log for reference.

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". Record the weights described in the procedure below in the Residue Determination Log (Attachment 1).

11.1 Preparing glass fiber filter discs: Place the disc on the membrane filter apparatus or insert it into the bottom of a suitable Gooch crucible. While the vacuum is applied, wash the disc with three successive 20 mL volumes of reagent water. Remove all traces of water by continuing to apply the vacuum after the water has passed through. Discard the washings.

11.2 Preparing evaporating dishes: If volatile residue is to be measured, heat the clean dishes to 550 °C for 1 hour in a muffle furnace. If only filterable residue is to be measured, heat the clean dishes to 180 °C ± 2 °C for 1 hour. Cool them in the desiccator and store until needed. Weigh immediately before use.

11.3 Assemble the filtering unit and begin suction while thoroughly mixing the sample (magnetic stirrer/TFR stirring bar**). Measure a 100 mL aliquot using a graduated cylinder and pour the** measured volume onto the glass-fiber filter. If the total filterable residue is low, a larger volume should be used. Sufficient volume of sample should be used to yield at least 2.5 mg of residue. Conversely, because excess residue in the dish may form a water-trapping crust, limit sample to no more than 200 mg. If more than 10 minutes are required to complete filtration, increase the filter size or decrease sample volume.

11.4 Filter the sample through the glass fiber filter and rinse with three 10 mL portions of reagent water allowing complete drainage between washes. Continue to vacuum for about 3 minutes after filtration is complete to remove as much water as possible.

11.5 Transfer the volume of the sample to a weighed evaporation dish and evaporate to dryness in a drying oven.

11.6 To avoid splattering, place the sample in the oven set at ~ 98 °C until the sample reaches dryness. Then increase the temperature to 180 °C, being careful not to exceed 180 °C. Dry the evaporated sample for at least 1 hr at 180 °C ± 2 °C and cool in a desiccator. Weigh the dried sample. Repeat the drying procedure until a constant weight is obtained or until the weight change is less than 4% of the previous weight or 0.5 mg. The drying procedure includes re-heating, re-desiccating, and re-weighing.

11.7 If the results are greater than 200 mg, a smaller volume must be filtered to obtain a result less than 200 mg. If the result is less than 2.5 mg, a larger volume, up to 1000 mL, must be filtered to obtain a result greater than 2.5 mg. If 1000 mL sample has less than 2.5 mg residue on the filter, the sample should be reported as < 2.5mg/L.

If an entire liter must be filtered for a sample designated by the client as QC, an additional liter bottle would be needed by the laboratory to perform the duplicate analysis.

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP 13.4, "Numerical Data Reduction".

12.1 Calculation of filterable residue concentration:

$$\text{filterable residue, mg/L} = \frac{(A-B) \times 1{,}000}{C}$$

where: A = weight of dish + residue in mg
B = weight of dish in mg
C = volume of sample in mL

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required. See SOP 12.1, "Hazardous Waste Disposal", regarding laboratory procedures for recycling solvent waste streams.

Samples preserved with HCl, HNO_3, or H_2SO_4 to pH <2 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Standards Methods for the Examination of Water and Wastewater, 20th Edition (1998), Method 2540C.

16.2 Standard Methods for the Examination of Water and Wastewater, 20th (1998) Editions, Method 1080

16.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

16.4 Quality Control SOP 13.4, "Numerical Data Reduction"

16.6 NELAC Standards, **effective July** 2003**, plus revisions**

16.7 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.8 CompuChem Quality Manual, Revision **13**, **November 17, 2009, plus revisons**

16.9 Department of Defense Quality Systems Manual for Environmental Laboratories, Version **4.1**, **4/22/09, plus revisions**

16.10 Quality Control SOP 13.17, "Analytical Balance Calibration and Maintenance"

17.0 Attachments as Tables, Diagrams and Flowcharts

17.1 Attachment 1 – **Example** Residue Determination Log

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Attachment 1

CompuChem
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Filterable Residue (TDS) SM 2540C

Worksheek No. 1 S(5) Rev.1

Analyst No.: _____ Date: _____ SDG #: _____

CompuChem ID	Sample Type	Filter ID Number	Initial Dish Weighing-First (g)	Initial Dish Weighing-Second (g)	Initial Dish Weighing-Third (g)	Sample Volume (mL)	Final Dish Weighing-First (g)	Final Dish Weighing-Second (g)	Final Dish Weighing-Third (g)	Net Residue (Final weight - dish) (g)	Results (net residue / sample vol) (mg/L)	Comment
				NA	NA	100		NA	NA	0.0000	0.00	

Balance Calibration (200.0000 g)	
Initial Weighing	g
Final Weighing	g
Tolerance +/- 0.2000 g	

Reviewed By: _____

Date: _____

Record of Temperature/Time	
Date samples in oven:	
Time samples in oven:	
Tempature of oven In:	
Date samples out of oven:	
Time samples out of oven:	
Temperature of oven out:	

LCS Reference
LCS: Mid Level Solids Standard
REF #

Shading has been applied to unused portions of this worksheet as a substitute for 'Z-out.

Constant Weight is obtained when the weight loss of successive weighting is less than 4 percent or less than 0.5 mg, whichever is less.

The presence of the Chemist's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist together with the chemist's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Balance used for weights: Denver Instr Serial #B034903 S class weights for balance calibration:Serial #NCSN050 Fisher Oven Serial #007O0173

jcf 080210

Note: This page subject to change without notice.

SOP 3.6.2.2

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501 Madison Avenue
Cary, NC 27513

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SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: IP 802 & 809 SOP Section #: 3.6.2.2 Revision #: 16

SOP Title: Analysis of Soil Samples for Total Organic Carbon (TOC) by SW-846 Method 9060A, Modified for Soils, and Lloyd Kahn

Effective date: (QA fills in) 2-9-11

- Procedure prepared by: Judith Dozier Date: 2/1/2011
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Date: 02-09-2011
- Reason for change: Addition of DoD procedures and protocols, Annual review
- This procedure meets the requirements of the following approved method references: SW-846, 3RD Ed., Update IIIB, "/04, Method 9060A, Modified for Soils and Lloyd Kahn Method, "Determination of Total Organic Carbon in Sediment" July 1988 modified for Soil DoD Quality Systems Manual for Environmental Laboratories, Version 4.2, 10/25/2010

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Instrument Procedure 802 and 809: Analysis of Soil Samples for Total Organic Carbon (TOC) by SW-846 Method 9060A, modified for Soils, and Lloyd Kahn

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Instrument Procedure 802 and 809: Analysis of Soil Samples for Total Organic Carbon (TOC) by SW-846 Method 9060A, modified for Soils, and Lloyd Kahn

1.0 Scope and Application

Soil and sediment samples are analyzed using the SSM-5000A Solid Sample Module that is interfaced with the TOC-VCSH analyzer. The instrument converts any organic carbon to carbon dioxide (CO_2) by catalytic combustion. The carbon dioxide is measured directly with a non-dispersive infrared detector.

A reporting limit of 100 mg/Kg is required for the Lloyd Kahn method.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of analysts experienced in the instrumentation or preparative methods and who have demonstrated the ability to generate acceptable results through QC samples and analyst capability studies.

2.0 Summary

TOC in soil is composed of a variety of organic compounds in various oxidation states. A high temperature combustion method is used to measure TOC in soil. The sample is acidified to remove inorganic carbon, and introduced to a combustion oven (900°C) where all carbonaceous matter is oxidized to carbon dioxide. The carbon dioxide is bubbled through an acidified liquid and then routed through a mist trap to remove water and scrub out any corrosive species formed. The gas is swept to a non-dispersive infrared detector which is specific for carbon dioxide measurement.

For the Lloyd Kahn method, one sample in a 20 sample batch is analyzed in quadruplicate. Quadruplicate analyses are required by Method 9060A. Perform quadruplicate analyses unless instructed otherwise by the client. When run in quadruplicate, both the average and the range are reported.

3.0 Definitions

3.1 Method detection limit (MDL) – The minimum concentration of an analyte that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte (40 CFR, Part 136, Appendix B)**. A minimum of seven sample replicates is required to calculate a statistical MDL. The MDL is an approximation of the DL. Any analyte concentration result at or above the MDL must also meet all qualitative identification criteria required by the test method in order to be reported as present.**

3.2 Detection Limit (DL) - The DL is the smallest analyte that can be demonstrated to be different from zero or a blank concentration at the 99 % level of confidence. The false positive rate at the DL is 1% and the false negative rate is 50%. Any analyte concentration result at or above the DL must also meet all qualitative identification criteria required by the test method in order to be reported as present.

3.3 Limit of Detection (LOD) – The LOD is the smallest concentration of an analyte that must be present in a sample in order to be detected at a 99% confidence level. The false negative rate at the LOD is 1 %. For DoD-QSM, non-detect sample results are reported as < LOD. Numeric values of measurements between the DL and the LOQ are reported as estimates. The DL < LOD ≤ LOQ.

3.4 Limit of Quantitation (LOQ) – The Limit of Quantitation is the lowest concentration of a substance that produces a quantitative result within specified limits of precision and bias. The LOQ must be within the calibration range.

3.5 Reporting Limit (RL) –

3.5.1 For Non-DoD work, the laboratory reporting limit is based on the lowest multipoint calibration standard concentration. For organic methods, values detected below the reporting limit and above the MDL may be reported and qualified as an estimated concentration.

3.5.2 The DoD-QSM defines the reporting limit as being the lowest concentration value specified by the client that meets project requirements for reporting data with known precision and bias for a specific analyte in a specific matrix. The LOQ cannot be greater than

the RL. Data reported below the RL must be flagged as estimated values if they are also less than the LOQ.

3.3 Reporting Units – mg/Kg

3.4 A **Sample Delivery Group (**SDG**)** is defined by the following, whichever is more frequent:

- each 20 field samples received within a case, or
- each 7 calendar day period during which field samples in a case are received (14 calendar days if requested by the client) beginning with the receipt of the first sample.

NOTE: The DoD-QSM and SC DHEC do not accept the SDG approach, unless the samples are prepared in a single batch. When a group of up to 20 field samples of a similar matrix are prepared as one batch, method-specified QC samples such as a method blank, laboratory control sample, matrix spike, matrix spike duplicate, and matrix duplicate must also be prepared together at a rate of 5% for DoD-QSM. If samples are batched together from different sites, project-specific QC must be processed.

3.5 DoD-QSM – Department of Defense Quality Systems Manual

3.6 SC DHEC – South Carolina Department of Health and Environmental Control

4.0 Interferences

4.1 Volatile organics can be lost during sample blending, particularly if temperature is allowed to rise.

4.2 Avoid contaminated glassware, plastic containers, and rubber tubing.

5.0 Safety

Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

Laboratory staff must review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

6.0 Equipment **and** Supplies

6.1 Total organic analyzer-- Shimadzu, TOC-VCSH, model # H51104335168

6.1.1 Automatic sample injector - ASI-V, model # H52104301622

6.1.2 Solid Sample Module – SMM-5000A, model # H525043

6.2 Analytical balance capable of weighing to 0.001g for weighing samples and preparation of standards

6.3 Glassware – Prepare by handwashing with a non-phosphate biodegradable detergent, then rinsing three times with DI water.

6.3.1 100 mL volumetric flasks

6.3.2 Volumetric pipettes

7.0 Reagents and Standards

Standards and reagent traceability information must be documented on the Standards/QC Sample Preparation for TOC Soil **work**sheet. (Attachment 1)

Note: **All reagents and standards must be ACS (American Chemical Society) grade or equivalent, unless otherwise denoted.** All standards and reagents are subject to change in vendor and in concentration. The Reporting Limits are also subject to change, but must remain at or above the lowest point in the calibration.

7.1 Reagent Water-All water used during preparation must be reagent-grade Type I with regard to resistivity of > 10 megohm-cm (18th and 19th Editions of Standard Methods, Method 1080), and referred throughout this SOP as DI water.

7.2 Carrier gas: purified oxygen, carbon dioxide free, and containing less than 1 ppm hydrocarbon.

7.3 Quartz Wool (Used as soil media for standards and instrument blank)

7.4 Sulfuric acid (20%)-reagent grade – Cautiously add 200 **mL** of concentrated sulfuric acid to a one liter volumetric containing approximately 500 mL DI water. After cooling, dilute to the mark with DI water. This solution is used to prepare the calibration standards.

7.5 Phosphoric acid – 1:1 by volume solution used for Lloyd Kahn procedure

7.6 10,000 ppm Stock Calibration Standard

7.6.1 Potassium hydrogen phthalate (KHP), reagent grade, 10,000 ppm. This aqueous standard is purchased from Tekmar-Dohrman and is used as the primary stock standard.

7.7 10,000 ppm Second Source Stock Standard used for the Initial Calibration Verification (ICV) and the Laboratory Control Sample (LCS).

7.7.1 The ICV (6000 ppm) is prepared by adding 60 **µ**L of the Second Source Stock Standard (10,000ppm) to a sample boat containing a portion of quarts wool.

7.7.2 The LCS (6000 ppm) is prepared at by adding 60 **µ**L of the Second Source Stock Standard (10,000 ppm) to a sample boat containing a portion of quarts wool.

7.8 5,000 ppm Continuing Calibration Verification (CCV) Standard

7.8.1 The concentration of the continuing calibration standard is 5,000 ppm. This standard is prepared by adding 50 µL of the 10,000 ppm stock standard to a sample boat containing a of portion quarts wool.

7.9 Matrix Spike

7.9.1 The matrix spike and matrix spike duplicate (MS/MSD) are spiked using the 10,000 ppm stock standard. Based upon the results of the original un-spiked sample, the MS/MSD samples are then spiked at 1 to 5 times the sample concentration.

8.0 <u>Sample Preservation and Storage</u>

8.1 Samples are preserved, and stored according to the tables in Sample Control SOP 4.1, "Receiving Samples" and 4.6, "Storing Samples." Sample holding times are also listed.

8.2 Soil samples are not preserved. They must be refrigerated at 4 °C ± 2 °C in amber glass containers with TFE lined caps. Samples must be analyzed within 28 days of sampling**, except when using the Lloyd Kahn method, which requires analysis within 14 days**.

9.0 Quality Control

9.1 The ICV is run immediately after the instrument is calibrated with a separate source KHP standard. Acceptance criteria are ± 10% of the true value.

9.1.1 If the ICV fails, reanalyze. If it still fails, re-calibrate and reanalyze.

9.2 An initial calibration blank (ICB) is run after the ICV. This result must not be greater than the reporting limit. If the result is above the reporting limit, repeat the calibration.

9.2.1 To meet the requirements of the DoD-QSM, the concentration of the analyte in the blank must be < one-half the reporting limit.

9.3 The method blank or preparation blank (PB) is prepared with each batch of not more than 20 field samples. The results of the method blank must not exceed the reporting limit. If the result is above the reporting limit, reanalyze the blank. If the result is still above the reporting limit, recalibrate and reanalyze.

9.3.1 The DoD-QSM requires that the content of the method blank must be less than one-half of the reporting limit.

9.4 The LCS is prepared with each preparation batch of not more than 20 field samples. The recovery in the LCS must be within ± 10% of the true value. If the LCS fails reanalyze. If is still fails, recalibrate and reanalyze the entire batch.

9.5 A CCV and continuing calibration blank (CCB) are run after every 10 samples and at the end of the sequence. Acceptance criteria are the same as the ICV/ICB. If the CCV or CCB fails, the previous 10 samples must be reanalyzed.

9.6 A MS/MSD are prepared (if requested by the client) in a batch of not more than 20 field samples. The spike recovery should be between 75 and 125% and a relative percent difference should be ≤ 20%. If the duplicate matrix spike acceptance criteria are not met, but the LCS is acceptable, a sample matrix effect is indicated and the batch is in control.

10.0 Calibration and Standardization

10.1 Prepare the sample boat for analysis by heating it in the TC furnace of the Shimadzu, TOC-VCSH TOC analyzer at 900 °C for 20 minutes.

10.2 Calibrate the instrument at five levels using the 10,000 ppm Stock Standard Solution (Section 7.6).

10.2.1 Create a calibration curve by selecting "new calibration curve" on the instruments PC.

10.2.2 Select the "ssm system" (soil sample module)

10.2.3 Select "edit cal points manually".

10.2.4 Name your calibration curve and select "linear regression"

10.2.5 Using Quartz Wool as the soil matrix for the standards, analyze the calibration curve at 0 (ICB), 25, 50, 75, and 100 uL of the 10,000 ppm Stock Solution (Section 7.6).

10.3 A correlation coefficient of ≥ 0.995 must be achieved in order for the calibration to be acceptable. (See Attachment 3 for an example Calibration Curve)

10.4 Verifying the Calibration

10.4.1 The initial calibration is verified by analyzing the 6000 ppm ICV standard and a blank or ICB. The ICV results must be within ± 10% of the true value and the ICB < the reporting limit. If the ICV fails, re-analyze. If the re-analysis, analyze a new initial calibration.

10.4.1.1 To meet the requirements of the DoD-QSM, the concentration of the analyte in the blank must be < one-half the reporting limit.

10.4.2 A 5000 ppm continuing calibration verification (CCV) standard and a continuing calibration blank (CCB) are analyzed, after every 10 samples, and at the end of the analytical sequence. The results of the CCV must be within ± 10% of the true value and the CCB < the reporting limit. If the CCV fails, reanalyze it once. If it still fails, the instrument must be

recalibrated and the previous samples since the last passing CCV/CCB must be reanalyzed.

10.5 To meet the requirements of the DoD-QSM, the concentration of the analyte in the blank must be < one-half the reporting limit.

10.6 Refer to the TOC-V Series SSM-5000A "Solid Sample Module for Total Organic Carbon Analyzer" Users Manual for instrument operation instructions for calibration.

10.7 Pipette Calibration

10.7.1 Ensure pipettes have been calibrated at the required frequency before use according to Inorganics SOP 9.1, "Calibrating Automatic Pipettes in the Inorganics Laboratory."

10.8 Balance Calibration

10.8.1 Ensure the balance has been calibrated for the day prior to use following Quality Control SOP 13.17, "Analytical Balance Calibration and Maintenance."

11.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures."

11.1 Discard any foreign objects such as sticks, stones, and leaves. Thoroughly mix the sample, reducing any clumps, with a metal spatula in a plastic weighing boat to create a homogeneous, representative sample.

11.2 Prepare the sample boat using the procedure in **Section** 10.1. Remove the sample boat; add a tuft of quartz wool, then place it on the analytical balance. Tare the balance and transfer 10 to 20 mg of soil sample to the boat.

11.3 Add one drop of 20% sulfuric acid to the top of the sample in the boat. For Lloyd Kahn samples, add 1:1 phosphoric acid. Allow the sample to stand at room temperature for 3-5 minutes or until effervescence ceases.

11.4 Place the sample boat into the hatch port. Close the hatch, and allow the detector baseline to stabilize. Press "run." Enter the sample ID, press enter. Enter the sample weight in mg, and press "start."

11.5 Slowly push the boat into the combustion tube during the period "inject now." When the run is complete, pull the boat to the cooling position..

11.6 After completion of the integration, select "next" for the next sample and repeat **Sections** 11.2 through 11.6.

11.6.1 One in twenty Lloyd Kahn samples is analyzed in quadruplicate. Both the average and the range are reported. A RSD of 20% must be achieved for quad samples or the analysis must be repeated.

11.6.2 Method 9060A requires samples to be analyzed in quadruplicate. Both the average and the range are reported.

11.6.3 Perform quadruplicate analyses unless instructed otherwise by the client. Both the average and the range are reported.

11.7 Refer to the TOC-V Series SSM-5000A "Solid Sample Module for Total Organic Carbon Analyzer" users manual for instrument operation instructions for sample analysis.

11.8 Calculating Dilutions

11.8.1 Determine a level of dilution that will result in a value within the upper half of the calibration range. This is an acceptable dilution.

11.8.2 For example, 7.0 mg silica gel mixed with 2.9 mg sample yields a dilution factor of 3.41. A 10 mg aliquot of this mixture is transferred to the boat. It should be recorded on the run log as "3.41x (7.0 mg silica gel + 2.9 mg sample)."

12.0 Data Analysis and Calculations

Calculations must be consistent with the Quality Control SOP13.4, "Numerical Data Reduction."

12.1 The Shimadzu, TOC-VCSH TOC Analyzer automatically calculates the concentration of organic carbon. When carbon is detected, an electronic peak is generated and integrated by the Shimadzu, TOC-VCSH computer where its area is calculated. The computer then plots the area of the peak on the calibration curve.

12.4 Calculation of the mean or average of a set of values:

$$\overline{X} = \frac{\sum_{i=1}^{n} X_i}{n}$$

where: n = total number of values
x_i = each individual value used to calculate the mean
x = the mean of n

12.5 Calculation of the standard deviation of a set of values:

$$Standard\ deviation = \sqrt{\frac{\sum_{i=1}^{n}\left(X_n - \overline{X}\right)^2}{n-1}}$$

12.6 Calculation of percent recovery:

12.6.1 LCS:

$$\%R = \frac{Amount\ found}{Amount\ spiked}\ x\,100$$

12.6.2 Matrix spikes:

$$\%\,R = \frac{Amount\ in\ spiked\ sample - Amount\ in\ unspiked\ (native)\ sample}{Amount\ spiked}\ x\,100$$

12.7 Calculation of % RSD

$$\%RSD = \left(\frac{Standard\ deviation}{\overline{X}}\right) \times 100$$

12.8 Calculation of RPD

$$RPD = \frac{|Value\ 1\ -\ Value\ 2|}{(Value\ 1\ +\ Value\ 2)/2}\ x100$$

13.0 Method Performance

This method was validated through in-house laboratory studies of method detection limits and precision and accuracy for single analyst. The data are retained by the QA department.

14.0 Pollution Prevention

The solvents used in this procedure pose little threat to the environment when recycled and managed properly. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best thing. See SOP 12.1, Hazardous Waste Disposal, regarding laboratory procedures for recycling solvent waste streams.

15.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with H_2SO_4 to pH <2 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

16.0 References

16.1 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846; 3rd Edition, Update IIIB, 11/04, Method 9060A, modified for soils, based on the TOC-V Series SSM-5000A "Solid Sample Module for Total Organic Carbon Analyzer" Users Manual.

16.2 Quality Control SOP, 13.6 "Proper Documentation Procedures"

16.3 Quality Control SOP, 13.4 "Numerical Data Reduction"

16.4 NELAC Standards, effective July 2003**, plus revisions**

16.5 QA/G6: Guidance for Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

16.6 CompuChem Quality Manual, Revision **13**, **November 17**, 2009, plus revisions

16.7 Lloyd Kahn Method, "Determination of Total Organic Carbon in Sediment," July 1988, modified for soils

16.8 Department of Defense Quality Systems Manual for Environmental Laboratories, Version 4.2, October 25, 2010

17.0 Attachments as Tables, Diagrams, and Flowcharts

17.1 **Example TOC** Standards/QC Sample Preparation **Worksheet**

17.2 Example Calibration Curve

Attachment 1

Standards/QC Preparation for Total Organic Carbon (TOC) in Soil

COMPUCHEM a Division of Liberty Analytical Corporation WORKSHEET 1 H (8) 1 Sequence Filename: _____

STANDARDS AND REAGENTS TRACEABILITY*	REF #:	MANUFACTURER	LOT#	EXPIRATION DATE
TOC Second Source Stock Standard (10,000 ppm)	1U(6)1-17-27	Science Stuff Inc.	9092427	11-12-012
TOC Calibration Source Stock CAL Standard (10,000 ppm)	1U(6)1-18-17	NSI Solutions	020910	02/2012
Concentrated Sulfuric Acid (H_2SO_4)	0A07003	Caledon	140517	02/2012
Concentrated Phosphoric Acid (H_3PO_4) (LLOYD KAHN only)	1U(6)1-20-2	EMD	50091021	8-13-2015

REAGENT/STANDARD LOT NUMBER**	PREPARATION INSTRUCTIONS
20%-H_2SO_4 – 01 12 11 jm	Add 20-mL of concentrated H_2SO_4 to 80-mL of reagent water
50%-H_3PO_4 – 01 12 11 jm	Add 20-mL of concentrated H_3PO_4 to 20-mL of reagent water in a 40-mL vial.
TOC ICV – 01 21 11 jm 01 22 11 jm (Second Source)	6,000 ppm; Pipet 60-µL of TOC Second Source Stock into the weigh boat (set volume to 100 ul)
TOC LCS – 01 21 11 jm (Second Source)	6,000 ppm; Pipet 60-µL of TOC Second Source Stock into the weigh boat. (set volume to 100 ul)
TOC CCV – 01 21 11 jm 01 22 11 jm	5,000 ppm; Pipet 50-µL of TOC Calibration Stock into the weigh boat. (set volume to 100 ul)
TOC Matrix Spikes yields N/A	TOC Stock 10,000 ppm Standard / sample weight(mg) x volume(uL) of spike solution = MS/MSD True value
LOQ TV = 100 ppm - 01 21 11 jm	Pipet 100-µL of TOC Calibration Stock cal std into the weigh boat (set volume to 10000 ul)
LOD TV = 75 ppm - 01 22 11 jm	Pipet 75-µL of TOC Calibration Stock cal std into the weigh boat. (set volume to 10000 ul)

*Ref# = Logbook ID, Page number, and item number from Materials Receipt Log
**Standard Lot Number consists of standard ID, date prepared, and initials of preparer.

The presence of the Chemist's/Analyst's employee ID number, or signature, on this run log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible chemist/analyst together with the chemist's/analyst's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation.

Standard Solutions Prepared and Entered By: _____ Date: _____

Reviewed By: _____ Date: _____

8/26/08:vr

Note: This page subject to change without notice.

Attachment 2

TOC-V Cal Curve Information
SSMCAL.2009_08_14_10_49_52.cal

Date of Creation 3:29:38 PM 8/15/2006
User
System SSM

Cal. Curve

Sample Name: CAL
Sample ID: 10000
Cal. Curve: SSMCAL.2009_08_14_10_49_52.cal
Status Completed
Comment:

Type	Anal.	Density
Standard	SSM-TC	1.000mg/uL

AbsC: 0.000ug

No.	Area	CNV	Abs C	Weight	Volume	Rem.	Ex.	Date / Time
1	0.000	0.000	0.000ug	0.000mg	0uL	*******		8/14/2009 11:29:51

Mean Area 0.000
Mean CNV 0.000
SD Area 0.000
CV Area 0.00%

AbsC: 250.0ug

No.	Area	CNV	Abs C	Weight	Volume	Rem.	Ex.	Date / Time
1	27.12	27.12	250.0ug	25.00mg	25uL	*******		8/14/2009 11:42:03

Mean Area 27.12
Mean CNV 27.12
SD Area 0.000
CV Area 0.00%

AbsC: 500.0ug

No.	Area	CNV	Abs C	Weight	Volume	Rem.	Ex.	Date / Time
1	53.60	53.60	500.0ug	50.00mg	50uL	*******		8/14/2009 11:54:27

Mean Area 53.60
Mean CNV 53.60
SD Area 0.000
CV Area 0.00%

AbsC: 750.0ug

No.	Area	CNV	Abs C	Weight	Volume	Rem.	Ex.	Date / Time
1	78.25	78.25	750.0ug	75.00mg	75uL	*******		8/14/2009 12:09:15

Mean Area 78.25
Mean CNV 78.25
SD Area 0.000
CV Area 0.00%

1/2

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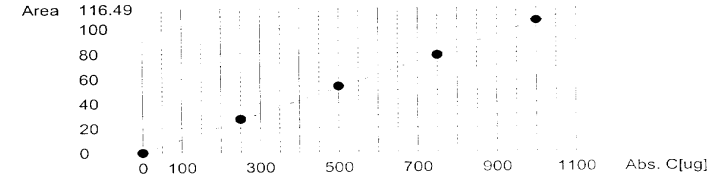
Attachment 2 (Continued)

AbsC: 1000ug

No.	Area	CNV	Abs C	Weight	Volume	Rem.	Ex.	Date / Time
1	105.9	105.9	1000ug	100.0mg	100uL	*******		8/14/2009 12:25:22 I

Mean Area	105.9
Mean CNV	105.9
SD Area	0.000
CV Area	0.00%

Slope:	0.1052
Intercept	0.000
r^2	0.999727
r	0.999864
Zero Shift	Yes



2/2

SOP 4.1

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure ✓ outdated procedure (archive) _____

- Procedure Code: _____ SOP Section #: 4.1 Revision #: 36

SOP Title: Receiving Samples

Effective date: (QA fills in) 07-14-10

- Procedure prepared by: Diane Byrd Date: 02-24-2010
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) Bob Meier Date: 07-12-2010
- Reason for change: Annual Review
- This procedure meets the requirements of the following approved method references: USEPA CLP SOW Documents SOMØ1.2 and ILMØ5.4; SW-846, 3RD Edition Update III 12/96 and Update IV 2/07; Code of Federal Regulations, Title 40, Part 136

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: [signature] Date: 4/4/11

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Sample Control SOP 4.1 Receiving Samples

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Sample Control SOP 4.1 Receiving Samples

1.0 Scope and Application

This Standard Operating Procedure (SOP) describes the process of receiving samples from the US EPA and commercial clients. It contains a full description of the log-in procedure.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

After all incoming samples have been received from the carrier at the loading dock, the Sample Control clerk **sorts the shipping containers, by client.** When **shipping containers are opened,** the sample containers must be checked to ensure that they are intact. Any damage to the sample containers must be noted on the appropriate paperwork. Chains-of-custody (COCs) must be verified against the actual sample received. Sample information is entered into the Laboratory Information Management System (LIMS) for scheduling and tracking purposes. Finally, the samples are placed into cold storage **or ambient storage** until preparation and analysis.

3.0 Definitions

3.1 **Sample Management Services Department - Incorporates the** "Receiving Department" or "Shipping and Receiving Department**.**" Staff **members** include the Supervisor of **Sample Management Services,** Sample Control Clerks, and the EPA Coordinator and/or their assistant.

3.2 LIMS – **Laboratory Information Management System**.

3.3 NJDEP - New Jersey Department of Environmental Protection

3.4 COC – a chain-of-custody form that arrives with the sample containers. It is located inside the cooler and it identifies the samples, the requested analyses, dates and signatures of people receiving and relinquishing custody of the samples,

the number of sample containers, etc.

3.5 Quality Assurance (QA) Notice - used to notify the client and laboratory of any outstanding issues or variances regarding the sample (e.g., sample temperature)

3.6 CompuChem Number (CCN) – A laboratory identifier assigned to each sample by the LIMS. The sample is tracked internally by means of the CCN.

3.7 Sample Control Clerk – designated staff who have responsibility for sample security, storage and custody and who control access to the sample storage units.

3.8 CLP Sample - U.S. EPA Contract Laboratory Program sample; refers to a sample received for analyses using a specified statement of work.

3.9 Commercial sample - refers to a non-CLP sample received for an analysis

3.10 Statement of Work (SOW) - a USEPA document that provides requirements about sample handling, preparation, and analysis.

3.11 CDP - **Client Documentation Package;** contains all of the documentation received with the sample containers in addition to forms completed during sample receipt. A copy of the CDP is stored in Customer Service by **Project Management.**

3.12 RAS - Routine Analytical Services signifies that the samples are to be processed exactly as presented in a specified SOW (i.e., routinely).

3.13 **Modified Analysis (MA) - An MA** identifies special preparation/analytical instructions not identified in an SOW but requested by the EPA **as part of organic and inorganic CLP SOW documents.**

3.14 CSF (Complete SDG File) Envelope – A metal clasp **envelope** that contains completed receiving documents. Originals are placed into the lowest numbered sample delivery group (SDG). One CSF envelope is generated for each water and soil organic and inorganic SDG. The CSF applies only to EPA unless requested by commercial clients.

3.15 Customer Service Department – The department includes Customer Service representatives and/or Project Managers depending on the current staffing requirements.

3.16 NCDENR – North Carolina Department of Environment and Natural Resources

3.17 VOA – volatile organic analysis

4.0 Safety

4.1 Appropriate protective equipment must be worn under the assumption that all samples are hazardous. At a minimum, safety glasses, gloves and lab coats must be worn when handling samples.

4.2 Laboratory staff members are required to review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment and Supplies

5.1 NIST – traceable thermometer with a minimum measurable range of 0 – 50 °C with a calibration tolerance of ± **1.0** °C.

5.1.1 Other temperature measurement devices that can be calibrated to ± **0.5** °C and have a range of 0 - 20 °C, such as an infrared (IR) gun.

5.2 **Narrow** range pH paper

5.3 **Lead** acetate test strips

5.4 **Potassium** iodide starch test strips

6.0 Sample Preservation and Storage

6.1 Attachment 1 contains the table of sample preservation and holding times.

6.2 See Sample Control 4.6, "Storing Samples" for storage conditions.

7.0 Quality Control

7.1 Should any problems or questions arise which are not adequately addressed in the text of this SOP, consult your supervisor.

7.2 Discrepancies noted during receipt and log-in are documented in Quality Assurance (QA) notices (Attachments 2 - 7 and 16), on the COC, or in communication logs and discussed with the client before proceeding. The **Project** Manager is notified of the discrepancy, contacts the client for resolution, and provides feedback to the Sample Control clerk on how to proceed.

7.3 All calibrated temperature measuring devices must be labeled with the calibration date, initials of the person performing the calibration, and have a unique identifier, i.e.**,** serial number.

7.4 Every attempt must be made to complete the sample receiving process so that samples do not remain outside refrigeration for longer than two hours. For samples put on hold or for which there are questions awaiting resolution from the client, put the samples on a cart and wheel them into the walk-in storage unit **or place them in Refrigerator #5, located in the Sample Receiving area,** until further notice.

8.0 Calibration and Standardization

8.1 The Vice President **and General Manager** (**VPGM**) calibrates the IR Gun every three months using the NIST thermometer. Sample Control staff **members** perform a check using three different bottle types (plastic, amber glass, and clear glass). A check of the calibration is **usually** performed **6 days a week** and is completed by placing a calibrated thermometer into each of the three different pre-chilled bottle types (water filled), and noting the temperature after the thermometer has stabilized. At the same time the thermometer stabilizes, the IR Gun should be placed approximately 13 inches from the bottle being checked and the trigger should be pulled to lock the temperature into the IR Gun's view screen. Each of the temperatures should be noted in **the** IR Gun Check **log (**Attachment 8). The tolerance for the check of the IR Gun is ± **0.5** °C.

8.2 The **VPGM** calibrates all NIST-traceable thermometers against a NIST thermometer on an annual basis. **Thermometers are calibrated at their point of use and the NIST thermometer is also calibrated at the ice point and in boiling water, depending on which thermometers are being calibrated.** Records are maintained in the Standards Laboratory. Temperatures of storage units are recorded daily in each of their respective logbooks (Attachment 9).

9.0 Procedure

Documentation must follow the requirements in Quality Control SOP 13.6, "Proper Documentation Procedures". The sample coolers are routinely delivered by standard courier such as Federal Express or UPS or may be hand-delivered from the field. All coolers are handled in the following manner.

9.1 Checking the cooler and each sample container for damage.

9.1.1 Check the exterior of the cooler for any signs of damage such as a broken **custody** seal, signs of sample leakage, etc. Note any observed damage on the commercial COC (Attachment 10) or EPA Organic/Inorganic Traffic Report (Attachments 11 and 12.)

9.1.2 Place the cooler under a hood to open. If this is not possible, place the coolers on the floor next to the hood. When removing the samples place them immediately in the hood.

9.1.2.1 Immediately after opening the cooler, and prior to the removal of any samples, the temperature of the sample shipping cooler is measured as described in Section 9.2 of this SOP.

9.1.2.2 If a sample container is completely broken, therefore unsalvageable, sample volume is properly disposed of following Hazardous Waste SOP 12.1, "Hazardous Waste Disposal".

9.1.2.3 If a sample container is broken or any type of sample spill occurs in the laboratory, clean-up must follow the guidelines given in Hazardous Waste SOP 12.2, "Spill Control and Cleanup". .

9.1.2.4 If a soil sample container is broken or cracked and the raw sample has not come into contact with another sample, the sample may be salvageable. The decision of whether to salvage the sample is left to the client. The Project Manager will notify the client of the situation. If the client chooses to continue the analysis, note this information on the COC.

9.2 Checking and recording the temperature of a sample.

9.2.1 Temperature Requirements at Sample Receipt

9.2.1.1 **For non-CLP clients and for** samples submitted to meet the regulatory requirements of North Carolina DENR, the temperature tolerance is **above freezing, but** ≤ 6 °C. For the CLP**,** the temperature must not exceed 10 °C. If the sample receipt temperature tolerances are exceeded, the client **[Sample Management Office (**SMO**)** for CLP samples**]** is contacted for guidance.

9.2.1.2 The NIST-traceable thermometer or IR gun must have a minimum measurable range of 0 – 50 °C with a calibration tolerance of ± 1.0 °C **for thermometers or ± 0.5 °C for the IR guns** in order to be used.

9.2.1.2.1 Alternate temperature measurement devices may be used if they can be calibrated to ± 1.0 °C and have a **minimum** working range of 0 **– 15** °C.

9.2.1.3 If a temperature reading is suspicious or does not meet the anticipated temperature range, the temperature should be double checked using an alternate measuring device. For example, if the IR gun returns a temperature below zero but the samples do not appear frozen or feel cold to the touch, then the temperature should be verified using a NIST-traceable thermometer or other measuring device.

9.2.1.3.1 If the problem persists, the measuring device should be removed from service until it is re-calibrated.

9.2.2 Temperature Measurement using the IR Gun

9.2.2.1 The IR Gun is used for temperature determination**s**. A temperature blank or other sample container (if no temperature blank is available) from the cooler (as close to the center as possible) is used. **If possible, t**he temperature must be measured upon opening the cooler and prior to unpacking the samples and removing any packing material.

9.2.2.2 Place the IR Gun approximately 13 inches from the bottle and squeeze the trigger. The bottle temperature will now be locked on the IR Gun's view screen. When aiming the IR Gun towards the bottle, eliminate as much background interference by holding the gun level and making sure that other material surrounding the bottle is not also being checked.

9.2.2.3 Record the temperature reading on the COC and log-in sheet. **Also record the IR gun identification number.**

9.2.3 Temperature measurement for Coolers received with a Temperature Indicator Bottle (temperature blank)

9.2.3.1 The temperature of the "USEPA Cooler Temperature Indicator,"

received under the USEPA CLP SOW, is measured with the IR gun using the procedure in **Section** 9.2.2. Some non-CLP clients refer to this temperature indicator as a temperature blank.

9.2.3.2 Alternatively, a calibrated NIST-traceable thermometer may be used. Remove the cooler temperature indicator bottle cap, insert the thermometer into the bottle, and allow a minimum of three minutes but not more than five minutes for equilibration before taking the temperature.

9.2.4 Temperature Measurement for Coolers received without a Temperature Indicator Bottle (temperature blank)

9.2.4.1 If a Cooler Temperature Indicator bottle is not present in an EPA cooler, the EPA Coordinator or Assistant is required to contact the SMO, inform them of that fact, and use an alternative means of determining the cooler temperature.

9.2.4.2 The following options are employed by CompuChem to determine the cooler temperature. As required by the CLP SOW documents, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative. Additionally, the QA Notice (**Attachments** 6 and 7) must be included for EPA samples.

- An aliquot from a sample bottle designated for extractable organics or metals is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after three minutes but not more than five minutes. The contents of the disposable container are then properly discarded.

- A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded**, along with the identification number of the IR gun.**

9.2.5 Recording Temperature Information

9.2.5.1 Record information on the receiving log (Attachment 13 for EPA organic samples and Attachment 14 for EPA inorganic samples) found in the Sample Control area. Also record the information on the EPA or commercial COC.

9.2.5.2 If the temperature is out of range (4 +/- 2 ^{o}C for most commercial clients except as stated below), complete the appropriate sections of the required QA Notice (Attachment 2). The Project Manager will notify the commercial client of unacceptable temperature readings so the client can direct CompuChem on how to process the samples.

If samples submitted to meet the regulatory requirements of the NCDENR are received at a temperature outside of the range specified by North Carolina (2 – 6 °C), CompuChem must notify the State Laboratory of the excursion.

9.2.5.3 If the Cooler temperature exceeds 10 °C, for EPA samples, the EPA Coordinator or Assistant contacts SMO and informs them of the temperature deviation. The SMO will obtain direction from the Region. This information is documented **through** e-mails which are included in the SDG Narrative section of the final report. The narrative information will list, by fraction, the EPA sample number of all samples received in a cooler with a temperature exceeding 10 ° C. The actual temperature should also be included in the narrative.

9.3 Verification Process for the COC

9.3.1 Remove all paperwork and each sample container from the cooler and place them on the receiving table under the hood.

9.3.2 **The Sample Control Clerk signs his/her** name and enter**s** the date and time (of delivery by courier) on the COC under "**Received By**."

9.3.3 Check each sample container for signs of damage. Note any observed damage on the COC**, on which the sample ID is listed.**

9.3.4 Verify that all information, including sample identifiers that appear on the sample container, matches the information on the COC, packing list, and traffic reports, which are located inside the cooler. Note any discrepancies on the COC. Contact the Customer Service representative or Project Manager for non-EPA samples so that they can contact the client for resolution.

9.3.5 If discrepancies are found, the EPA Coordinator or Assistant notifies the SMO. The EPA Coordinator or Assistant must note the discrepancy on the Traffic Report.

9.3.6 If the following conditions are met, on each complete EPA Traffic Report, stamp or write the statement "received in good condition" or for commercial clients circle the "Y"/"N" option on the receiving log and initial and date (mm/dd/yy).

- all sample containers were received intact
- all information on the COC represents actual Cooler contents
- all associated sample tags were present (EPA sample only)
- all custody seals were intact
- all inorganic and wet chemistry samples were at the proper pH
- all corresponding documentation was present

Note: Sample temperature is not a consideration when using the statement "received in good condition." However, the temperature must be noted on the receiving log and the Traffic Report/COC.

9.3.7 If the samples do not meet the "good condition" criteria established above, make the proper notations on the COC and the receiving log, and initial and date the notations.

9.4 Measuring sample pH

9.4.1 Check the pH of every pre-preserved inorganic **and** wet chemistry sample, following Sample Control SOP 4.3, "Checking and Recording pH of Aqueous Cyanides; Phenols; and Wet Chemistry Samples."

9.4.2 Record the pH of the samples checked on the receiving log under the pH column, and on the COC. If a sample is not pre-preserved or is a VOA or GRO that cannot be opened due to headspace, record "NA" under the pH column of the receiving log.

9.4.3 If the pH is out of range, complete the appropriate QA notice (Attachment 3). The Project Manager will notify the commercial client of unacceptable pH readings so the client can direct the laboratory on how to process the samples. The EPA Coordinator or Assistant will contact SMO.

9.4.3.1 In those situations where the client requests the laboratory to preserve samples in-house, the lot number of any preservative used is added to the appropriate Quality Assurance Notice (QAN-R-2). See Attachment 3.

9.4.3.2 In those situations where nitric acid (50%) is required to be added to improperly preserved samples submitted for metal/mercury analysis to achieve a pH of < 2.0, a 24-hour waiting period has to occur before analysis can take place.

9.4.4 For samples received which are improperly preserved and have been submitted to meet the regulatory requirements of the NC DENR, the laboratory is responsible for notifying the State Laboratory of the situation. If a replacement sample cannot be obtained, results of the original sample must be qualified.

9.5 Residual chlorine and sulfide checks

9.5.1 Perform a residual chlorine check on all samples that are being analyzed for cyanide and phenol and perform a sulfide check for all samples to be analyzed for cyanide. See Sample Control SOP 4.13, "Handling and Verifying Proper Preservation of Samples Being Analyzed for Cyanide or Phenol". Fill out the appropriate QA Notice (Attachment 4 and Attachment 5)**,** if needed.

9.6 Volatile Organic Samples

9.6.1 Check each volatile water sample for air bubbles and headspace (empty space inside the vials) by inverting the 40 mL vial. Note any observance of air bubbles or headspace on the COC and the receiving log. Air bubbles larger than the size of a pea should be brought to the attention of the Customer Service representative or Project Manager so that the commercial client can be contacted to obtain instructions on how to proceed. The EPA Coordinator or Assistant will contact SMO.

Note: Pea size bubbles are those not exceeding ¼ inch or 6 mm in diameter. This is defined in SW-846 Chapter 4, Update III, 12/96.

For NJDEP aqueous VOA samples, no air bubbles or headspace is allowed.

9.6.2 In instances where aqueous samples have been submitted to meet the regulatory requirements of NCDENR, the State Laboratory must be

notified if there is headspace or large air bubbles. If no replacement sample can be obtained, the results of the original sample must be qualified.

9.6.3 Volatile soil samples received in EnCore™ samplers or glass jars must be delivered to the volatile lab immediately after log-in for preservation by the volatile lab staff.

9.7 Log-in for commercial samples

9.7.1 Record all receiving information (including but not limited **to** sample pH, cooler temperature, discrepancies) on the client COC, for commercial cooler receipts.

9.7.2 The Sample Control **Clerk** forwards the paperwork to the Project Manager where the following sample receiving steps are completed.

9.7.2.1 The commercial client is contacted, if necessary, regarding any discrepancies noted upon sample receipt.

9.7.2.2 The Project Manager, Sample Control Clerk, or Supervisor of **Sample Management Services** performs the order entry procedure at which point CCNs are assigned. See Customer Service SOP 5.11, "Order Entry in LIMS."

9.7.2.3 Project Managers maintain the **Client Documentation Package** (CDP). A copy of the CDP is scanned as an Adobe Acrobat .pdf file and maintained electronically for reference.

9.7.2.4 The CDP contains "original" paperwork and COCs and are maintained in client/project specific files in Customer Service/Project Management. Client-specific requirements are met for handling of "original" paperwork.

9.7.3 At the request of the commercial client, volatile holding/storage blanks are created as described in Sample Control SOP 4.9, "Preparing and Handling Test Samples." Test samples are routinely generated for EPA samples.

9.7.4 After order entry, bottle labels are printed from the LIMS to the printer in the Receiving **Department**. **The Sample Control Clerk records information from the generated labels into the Receipt/Storage Log (Attachment 17). The** Sample Control Clerk labels all sample containers with the laboratory-generated labels, leaving as much of the original field

label exposed as is possible, particularly the field sample ID. The field sample ID must be carefully checked against the lab-generated label at the time the labels are placed on the containers.

9.7.4.1 The Project Manager must communicate information pertaining to samples designed for long-term storage.

9.7.4.2 The Project Manager must communicate information pertaining to samples arriving from outside of the continental U.S, and designated quarantine states within the continental U.S. (NC, SC, GA, FL, TN, AR, OK, TX, LA, MS, AL**, NM, AZ, CA, NY**), **if that information is known. A piece of red and white striped tape is affixed to each sample bottle received from the quarantine areas for ease of identification.**

9.7.5 The labeled samples are placed onto a cart and moved into the secured cooler. Water samples not associated with US EPA CLP SOW ILM05.4 to be analyzed for total and dissolved metals are moved into the secured ambient storage room unless hexavalent chromium is requested.

9.7.5.1 Samples submitted, in order to meet the regulatory requirements of the NCDENR, must be maintained at 2 - 6° C, except for aqueous, metals-only samples. For that reason, those samples must be stored in a refrigerator that consistently has been maintained at equal to or less than 6° C but greater than or equal to 2° C.

9.8 Log-in for EPA samples

Note: Separate receiving logs are required for inorganics and organics and for water and soil matrices.

9.8.1 The Sample Control **Clerk** completes all areas of the receiving log except for the following areas that are completed by the EPA Coordinator or Assistant.

- Login No./SDG
- Contract No.
- Case No.
- TAT

- Lab Instructions
- $ Amount
- Sample No.
- QC
- Sample Login by

9.8.2 The paperwork is provided to the EPA Coordinator or Assistant who must immediately complete the receiving documentation required for the CSF including the Form DC-1 (Attachment 15). Those steps are detailed in Customer Service SOP 5.8, "Creating the Gray Folder." The EPA Coordinator or Assistant also enters the sample receipt information into the LIMS.

9.8.3 This includes the generation of bottle labels.

Note: A **Form** DC-1 must be completed for each cooler containing EPA samples

9.8.4 For EPA CLP dissolved metals samples, review the traffic report or COC for digestion instructions. If there are none, the samples designated as dissolved metals will be digested.

9.8.5 Once labels are generated, the Receiving Clerk wraps the appropriate label onto each sample container leaving the field sample ID that appears on the existing label in view, if possible.

9.8.**5**.1 If the samples are designated for long-term storage, as stated on the COC or by the SMO, place an additional label with the disposal date noted, on each container**, or on the storage box containing multiple samples having the same disposal date.**

9.8.5 Create volatile holding/storage blanks at the rate of two per SDG and place them in the volatile coolers with the samples. Blanks are created as described in Sample Control SOP 4.9, "Preparing and Handling Test Samples."

9.8.6 Place all labeled samples on a cart and move the samples into the appropriate secured cooler or ambient storage unit.

9.9 Contingency

9.9.1 If, due to a lab accident or to QC failure, a re-preparation is required for the sample and insufficient volume remains, the Project Manager must be alerted and will contact the client for direction on how to proceed.

9.9.2 If persistent contamination occurs in the laboratory, analyses must be halted until the source can be identified and isolated. When the contamination issue is resolved, sample analyses may proceed.

9.9.3 Refer to the corresponding Data Review SOP (number will vary among sections) for information on how to handle reporting of data that are unacceptable or out-of-control.

9.9.4 Any other issues that potentially affect data quality should be addressed with the Project Manager.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3 or H_2SO_4 to pH <2 or NaOH to pH > 12 are hazardous and must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab.

11.0 References

11.1 U.S. EPA CLP SOW **Documents** SOM01.2, and ILM05.4

11.2 "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods", SW-846, 3rd Edition, Update III, 12/96 **and Update IV, 2/07**

11.3 Quality Control SOP 13.6, "Proper Documentation Procedures"

11.4 NELAC Standards, effective July 2003**, plus additions**

11.5 EPA QA-G6: Guidance for the Preparing Standard Operating Procedures (SOPs) EPA/600/B-07/001, April 2007.

11.6 CompuChem Quality Manual, **Revision 13, November 17, 2009, plus revisions**

12.0 Attachments as Tables, Diagrams and Flowcharts

Attachment 1 – Tables of Preservation and Holding Times **(Taken from CompuChem Quality Manual, Revision 13, November 17, 2009, plus revisions)**

Attachment 2 – Quality Assurance Notices for Temperature Excursion

Attachment 3 – Quality Assurance Notice for pH

Attachment 4 – Quality Assurance Notice for Cyanide Samples

Attachment 5 – Quality Assurance Notice for Phenol **Samples**

Attachment 6 – Quality Assurance Notice for Cooler Temperature Options – Organics

Attachment 7 – Quality Assurance Notice for Cooler Temperature Options – Inorganics

Attachment 8 – Logbook 7 S**S** 1, IR Gun Check

Attachment 9 – Temperature Log Book Example

Attachment 10 – Chain-of-Custody Record

Attachment 11 – EPA Organic Traffic Report Example

Attachment 12 – EPA Inorganic Traffic Report Example

Attachment 13 – EPA Receiving Log - **Organic**

Attachment 14 – EPA Receiving Log - **Inorganic**

Attachment 15 – **Sample Log-In Sheet** (Form DC-1), Organic & Inorganic

Attachment 16 – QA Notices for North Carolina Only

Attachment 17 – Sample Receipt/Storage Log

Attachment 1

Table 4.1

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the Clean Water Act, 40 CFR 136, ***Federal Register, March 12, 2007***

Parameter	Preservation	Holding[a] Time (days)	Containers[b]	Volume mL
Acidity	Cool, ≤ 6 °C[g]	14	P or G	200
Alkalinity	Cool, ≤ 6 °C[g]	14	P or G	100
Ammonia	Cool, ≤ 6 °C[g], H_2SO_4 to pH<2	28	P or G	500
Biochemical Oxygen Demand	Cool, ≤ 6 °C[g]	48 hours	P or G	1000
Bromide	NR[c]	28	P or G	200
Chemical Oxygen Demand	Cool, ≤ 6 ° C[g], H_2SO_4 to pH<2	28	P or G	100
Chloride	NR	28	P or G	100
Chlorine, Total Residual	NR	0[d]	P or G	500
Chromium VI	Cool, ≤ 6 °C[g]	24 hours	P or G	500
Coliform, fecal and total	Cool, < 10 °C; 0.008% $Na_2S_2O_3$	6 hours	P or G	200
Color	Cool, ≤ 6 °C[g]	48 hours	P or G	500
Cyanide, total	Cool, ≤ 6 °C[g]; NaOH to pH>12 0.6 g ascorbic acid[f]	14[e]	P or G	1000
Cyanide, amenable to chlorination (free)	Cool, ≤ 6 °C[g]; NaOH to pH>12 0.6 g ascorbic acid[f]	14[e]	P or G	500
Fluoride	NR	28	P	500
Hardness	HNO_3 to pH<2 H_2SO_4 to pH <2	180	P or G	250
Hydrogen Ion (pH)	NR[c]	0[d]	P or G	40
Kjeldahl Nitrogen	Cool, ≤ 6 °C[g]; H_2SO_4 to pH <2	28	P or G	1000
Mercury	HNO_3 to pH<2	28	P or G	500
Metals (except Cr VI and Hg)	HNO_3 to pH<2	180	P or G	500
Nitrate (as N)	Cool, ≤ 6 °C[g]	48 hours	P or G	100
Nitrite (as N)	Cool, ≤ 6 °C[g]	48 hours	P or G	50
Nitrate-Nitrite (as N)	Cool, ≤ 6 °C[g]; H_2SO_4 to pH <2	28	P or G	500
Oil and Grease	Cool, ≤ 6° C[g]; H_2SO_4 to pH <2	28	G	1000
Organic Carbon, Total (TOC)	Cool, ≤ 6 °C[g] HCl or H_2SO_4 to pH <2	28	P or G	100
Organic Nitrogen, Total	Cool, ≤ 6°C[g]; H_2SO_4 to pH <2	28	P or G	500
Orthosphosphate (as P)	Filter immediately, Cool, ≤ 6 °C[g]	48 hours	P or G	50
Phenols (Total)	Cool, ≤ 6°C[g]; H_2SO_4 to pH <2	28	G	1000
Phosphorus (Elemental)	Cool, ≤ 6 °C[g]	48 hours	G	500
Phosphorus (Total)	Cool, ≤ 6°C[g] H_2SO_4 to pH <2	28	P or G	200

[a] From time of sample collection
[b] Polyethylene (P) or glass (G)
[c] None required
[d] 0 days indicates that the sample must be analyzed immediately.
[e] Reduced to 24 hours if sulfide is present, unless sulfide is removed before preservation.
[f] Added if residual chlorine is present
[g] Aqueous samples must be preserved at ≤ 6 °C, and should not be frozen unless data demonstrating that sample freezing does not adversely impact sample integrity is maintained on file and accepted as valid by the regulating authority. Also, for purposes of NPDES monitoring, the specification of "≤ °C" is used in place of the "4 °C" and "< 4 °C" sample temperature requirements listed in some methods.

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Attachment 1 (Continued)

Table 4.1 (Continued)

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the Clean Water Act, 40 CFR 136, *Federal Register, March 12, 2007*

Parameter	Preservation	Holding Time[a] (days)	Containers[b]	Volume (mL)
Solids (Total and Filterable)	Cool, ≤ 6 °C	7	P or G	100
Solids (Non-filterable)	Cool, ≤ 6 °C	7	P or G	300
Solids (Settleable)	Cool, ≤ 6 °C	7 days	P or G	1000
Silica	Cool, ≤ 6 °C	28	P	100
Specific Conductance	Cool, ≤ 6 °C	28	P or G	250
Sulfate	Cool, ≤ 6 °C	28	P or G	250
Sulfide	Cool, ≤ 6 °C Add zinc acetate and NaOH to pH>9.	7	P or G	500
Sulfite	NR[c]	0[d]	P or G	250
Surfactants	Cool, ≤ 6 °C	48 hours	P or G	250
Turbidity	Cool, ≤ 6 °C	48 hours	P or G	250
Purgeable Halocarbons	Cool, ≤ 6 °C 0.008% $Na_2S_2O_3$[e]	14	G	80
Purgeable Aromatic Hydrocarbons	Cool, ≤ 6 °C 0.008% $Na_2S_2O_3$[e] HCl to pH<2	14	G	80
Phenols	Cool, ≤ 6 °C 0.008% $Na_2S_2O_3$[e]	7/40[f]	P or G	2000
Benzidines	Cool, ≤ 6 °C 0.008% $Na_2S_2O_3$[e]	7/7[g]	P or G	2000
Phthalate Esters	Cool, ≤ 6 °C	7/40[f]	G	2000
Nitrosamines	Cool, ≤ 6 °C, dark 0.008% $Na_2S_2O_3$[e]	7/40[f]	P or G	2000
PCBs	Cool, ≤ 6 °C	7/40[f]	P or G	2000
Nitroaromatics and Isophorone	Cool, ≤ 6 °C, dark 0.008% $Na_2S_2O_3$[e]	7/40[f]	P or G	2000
Polynuclear Aromatic Hydrocarbons	Cool, ≤ 6 °C, dark 0.008% $Na_2S_2O_3$[e]	7/40[f]	G	2000
Haloethers	Cool, ≤ 6 °C 0.008% $Na_2S_2O_3$[e]	7/40[f]	P or G	2000
Chlorinated Hydrocarbons	Cool, ≤ 6 °C	7/40[f]	P or G	2000
TCDD	Cool, ≤ 6 °C 0.008% $Na_2S_2O_3$[e]	7/40[f]	P or G	2000
Pesticides	Cool, ≤ 6 °C; pH 5-9	7/40[f]	P or G	2000

[a] From time of sample collection
[b] Polyethylene (P) or glass (G)
[c] None required
[d] 0 days indicates that the sample must be analyzed immediately.
[e] $Na_2S_2O_3$ added if residual chlorine is present; approx. equal to 1 mL of 10% thiosulfate/liter of sample.
[f] To complete extraction (or initiate extraction if continuous liquid-liquid extraction is performed)/ to complete analysis following extraction
[g] Extracts may be stored up to 7 days before analysis if stored under an inert (oxidant-free) atmosphere.

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Attachment 1 (Continued)

Table 4.2

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the U.S. EPA CLP SOW for Inorganics Analysis, ILM04.1/ILM05.4

Parameter	Preservation[a]	Holding Time[b] (days)	Containers[c]	Volume (mL)
Cyanide, total	Cool, 4 °C; NaOH to pH>12 0.6 g of ascorbic acid[d]	12	P or G	1000
Metals (except Hg)	HNO_3 to pH<2, 4 ° ± 2 °C	180	P or G	500[e]
Mercury	HNO_3 to pH<2, 4 ° ± 2 °C	26	P or G	500[e]

a Water sample only; preservation performed by sampler immediately upon sample collection. Soil/sediment samples are maintained at 4 °C until analysis. Dissolved metals are filtered on site by sampler before addition of preservative. Water samples received under the ILM04.1 SOW are stored at ambient temperature
b From validated time of sample receipt
c Polyethylene (P) or glass (G)
d Only used in the presence of residual chlorine
e Can be combined into a one-liter bottle.

Table 4.3

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes Published in the U.S. EPA CLP SOW for Organics Analysis OLM04.3, and OLC03.2 (aqueous only), and SOM01.2

Parameter	Preservation	Holding Time (days)[a]	Containers[b]	Volume (mL)
Aqueous Volatiles	Cool, 4 ° ± 2 °C[e] HCl to pH≤2	10[d]	G-TLSSL	80
Soil/Sediment Volatiles	4 ° ± 2°C[c], sodium bisulfate < -7°C	10[d] 10	G-TLC, CET, EnCore™ samplers	4 oz 60 mL
Aqueous Semivolatiles	4 ° ± 2 °C[c]	5/40[e]	AG	2000
Soil/Sediment Semivolatiles	4 ° ± 2 °C[c]	10/40[e]	G	8 oz
Aqueous Pesticides/PCBs	4 ° ± 2 °C[c]	5/40[e]	AG	2000
Soil/Sediment Pesticides/PCBs	4 °± 2 °C[c]	10/40[e]	G	8 oz

[a] From validated time of sample receipt
[b] All containers are 1-liter amber glass (AG) bottles with Teflon-lined cap except aqueous volatiles (G-TLSSL = 40-mL glass bottle with Teflon-lined septum sealed lid), and soil/sediment volatiles (G-TLC = 4-oz glass jar with Teflon-lined cap or CET = closed-end tubes such as brass sleeves). Soil samples may also be collected under OLM04.3 and SOM01.1 for low level analysis in Encore™ samplers or as 5 gm sample in sodium bisulfate or medium level analysis in pre-weighed vials containing 10 mL methanol. When this occurs, the sample vial, with 10 mL methanol and all labeling, is weighed to the nearest 0.1 g prior to the addition of sample. Approximately 5 g of sample is added to the vial. The sample vial with sample is weighed to the nearest 0.1 g. The initial weight, final weight and sample weight are recorded and provided to the laboratory.
[c] Preserve samples at time of collection; samples should be stored in the dark until extraction/analysis.
[d] Until analysis
[e] To complete extraction/to complete analysis following extraction

Attachment 1 (Continued)

Table 4.4

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Aqueous and Solid Matrices[a] Published in SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Update IV, February 2007.

Parameter	Methods	References	Container	Preservation	Maximum Holding Times[b]
Volatile Organics in water	Purge and trap GC and GC/MS	8015A modified/ 8015B, 8015C 8260B	Glass, 40-mL vial with zero headspace	Cool to ≤ 6 °C, HCl to pH <2, 0.008% $Na_2S_2O_3$	14 days (A) (See below for soil)
Semivolatile Organics	GC, HPLC, and GC/MS	8081A, 8081B, 8082, 8082A, 8151A, 8310, 8270C, 8270D, 8330, 8330A, 8332	Glass, amber (A) (1 liter sample); 8-oz wide mouth with PTFE-lined cap (S) (50-g min. ea. test)	Cool to ≤ 6 °C, 0.008% $Na_2S_2O_3$	7 days to extract (A); 14 days to extract (S); 40 days to analyze (A/S)
Total Metals – except Mercury and Chromium VI	ICP AES, ICP/MS	6010B, 6010C, 6020, 6020A	Glass or plastic 500 mL (A), 8-oz wide mouth (S) (200-g sample)	HNO_3 to pH<2 (A); filter dissolved on-site first; filter suspended-no acid	6 months (A/S)
Chromium VI	Colorimetric, automated	7196A	Glass or plastic, 500 mL (A), 8-oz wide mouth (S) (200-g sample)	Cool to ≤ 6 °C	24 hours (A); one mo. to extraction, 4 days after (S).
Mercury	Manual cold vapor AA	7470 7471A 7471B	Glass or plastic, 500 mL (A), 8-oz wide mouth (200-g sample)	pH<2 HNO_3 (A); Cool to 4C (S)	28 days (A/S)
Cyanide	Colorimetric automated	9012A	Glass or plastic	Cool to ≤ 6 °C; pH >12 NaOH (A)	14days (A)
Phenols	Colorimetric, automated	420.2	Glass or plastic	Cool to ≤ 6 °C; pH < 4 H_2SO_4 (A)	28 days (A)
Specific conductance	Conductivity	9050A	Glass or plastic	NA	28 days (A)
Sulfide	Distillation, titration	9034	Glass or plastic	Cool to ≤ 6 °C; Zinc acetate (A/S)	7 days (A/S)
Oil & grease	Gravimetric	9070 9071A	Glass	Cool to 4C (A/S); 5 ml diluted HCl(A)	28 days (A) 14 days (S)
TOC	Combustion analyzer	9060	Glass or plastic	Cool to ≤ 6C; pH<2 HCl or H_2SO_4; Store in dark (A)	28 days

[a] Table originally excerpted, in part, from Table II, 40 CFR 136 Vol. 72, No. 47/Monday, March 12, 2007/Rules and Regulations.
b Holding time begins at time of sample collection.
[c] $Na_2S_2O_3$ added if residual chlorine is present; approx. equal to 1 ml of 10% thiosulfate / liter of sample.
(A) aqueous, (S) solid

ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 1 (Continued)

Table 4.5

Sample Handling, Preservation and Holding Time Table for SW-846 Method 5035, Volatile Organics in Soil

Conc. Level	Sampling Device	Collection Procedure	Container Type	Container Preparation	Preservation	Maximum Holding Time[a]
≤ 200 µg/kg	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1	Cool, ≤ 6°C, $NaHSO_4$	14 days
	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1[b]	Cool, ≤ 6 °C	48 hours
	Coring Device	5035-Section 6.2.1	Glass vial w/ PTFE-silicon septum	5035-6.1.1	Cool, ≤ 6 °C/-10°C[d]	48 hours/ 14 days[e]
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[b,f,g]	Cool, ≤ 6 °C	48 hours
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[f,g]	$NaHSO_4$, Cool, ≤ 6 °C	48 hours, 14 days
	Encore™ or equivalent	5035-Section 6.2.1	Encore™ or equivalent	5035-6.1.1[b,f,g]	Cool, ≤ 6 °C /-10 °C[c,d]	48 hours/ 14 days
> 200 µg/kg[h]	Encore™ or equivalent	5035-Section 6.2.2.3[f]	Encore™ or equivalent	5035-6.1.1[f,g]	Cool, ≤ 6 °C	48 hours 14 days
	Coring Device	5035-Section 6.2.2.3[I]	Glass vial w/ PTFE-silicon septum	5035-6.1.1[i]	Methanol/PEG Cool, ≤ 6 °C	14 days
	Conventional Devices	FLDEP SOP –Section 4.3	Glass vial w/ PTFE-silicon septum	5035-6.1.1	Cool, ≤ 6 °C	14 days
Dry weight	Conventional Devices		Glass w/ teflon liner		Cool, ≤ 6 °C	

[a] Maximum time allowed from time/date of collection to sample analysis.
[b] Eliminate 6.1.1.2; use only organic-free water.
[c] Contents of sampling device must be transported to the laboratory at ≤ 6 °C and stored at –10 °C; this option upon client request.
[d] In order to ensure that vials do not break during freezing, they should be stored on their side or at a slanted angle.
[e] Maximum allowed time at ≤ 6 °C is 48 hours; maximum allowed time to sample analysis is 14 days from collection.
[f] Conducted in the laboratory.
[g] Entire contents of sampling device is extruded into the sample analysis vial containing the appropriate solvent.
[h] Procedures are limited only to those situations or programs in which the maximum contamination level does not exceed 200 µg/kg.
[i] Methanolic preservation in the field is not recommended.

Attachment 1 (Continued)

Table 4.6

Requirements for Holding Times for Hazardous Characteristics Indicators Published in SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Update 3, December 1996.

Parameter	Methods	References	Container	Preservation	Maximum Holding Times
Reactivity-Total releasable cyanide	Reflux distillation, colorimetry	Chapter 7, 7.3.3, 9014	Glass, with zero headspace	≤ 6C in dark, pH>12 NaOH	ASAP
Reactivity-Total releasable Sulfide	Acid distillation, titration	Chapter 7, 7.3.4, 9034	Glass, with zero headspace	≤ 6C in dark, pH>9 NaOH; zinc acetate	ASAP
Ignitability	Flash point	1010	Glass or plastic	NA	ASAP
Corrosivity	Electrometric	9040B	Glass or plastic	NA	ASAP
Paint filter liquids	filtration	9095A	Glass or plastic	NA	ASAP

Table 4.7

Requirements for Containers, Preservation, Holding Times, and Recommended Sample Volumes for Toxicity Characteristics Leaching Procedure, TCLP, Method 1311, and Synthetic Precipitation Leaching Procedure, SPLP, Method 1312, Published in SW-846 Test Methods for Evaluating Solid Waste, Third Edition, Update III, December 1996 and Update IV, February 2007.

Sample Maximum Holding Times				
Parameter	**From: Field collection To: TCLP Extraction**	**From: TCLP Extraction To: Preparative Extraction**	**From: Preparative Extraction To: Determinative Analysis**	**Total elapsed time in days**
Volatiles	14	NA	14	28
Semivolatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, no Hg	180	NA	180	360

Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

Holding time associated with the ASTM leachate generation method (D3987-85) is 14 days from collection to leachate generation, i.e. filtration, then the method holding time to preparation and/or analysis.

Attachment 2

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE

Client _____
Case # _____
Type of Analysis _____
Receipt Date _____

For some organic and/or inorganic determinations temperature preservation at 4 degrees Celsius is required for environmental samples during shipment to the laboratory and prior to analysis. A temperature tolerance range is generally allowed. Temperature of a representative sample from the shipping container is taken and recorded by the receiving clerk at the time of sample receipt. This temperature is representative of all samples contained in the cooler.

The EPA CLP program requires the laboratory make notification when the temperature exceeds 10° C.

The State of North Carolina requires that samples must be iced to above freezing but ≤ 6° C during shipment.

Notification to other clients is either client or project dependent.

Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

The temperature of this sample at the time of receipt was determined to be _____.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Hand Delivery/Received on ice _____

Analyze - qualify with notice _____

Dispose - client will resample _____

Supervisor Signature/ID _____/_____ Date _____

QAN-R-3
100319

qanr3 – 3/19/10:jad

Attachment 3

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is_____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ Date _____

Preservation Type_____ Preservative Lot Number_____

Preserved By_____ Date_____

QAN-R-2
100319

QAN-R-2:031910:jad

Attachment 4

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

A chlorine and sulfide check was performed on the above cyanide sample

The results are checked below.

Chlorine was detected _____
Sulfide was detected _____

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

Dispose - client will resample _____

Supervisor Signature/ID _____/_____

Date _____

QAN-R-1
100319

Qanr1 – 3/19/10:jad

Attachment 5

CompuChem
A Division Of
Liberty Analytical Corp.

Quality Assurance Notice

CompuChem ID#_____
Client ID#_____
Case #_____
SDG# _____
Receipt Date_____
Method_____

A chlorine check was performed on the above phenol sample and was determined to be present. A member of CompuChem's Customer Service Department contacted the client. The Receiving Department was instructed as follows:

Analyze – Qualify with notice and address in narrative: _____
Dispose – Client will resample: _____

Supervisor Signature/ID _____/_____

Date _____

QAN-R-4
100319

Qanr4 – 3/19/10:jad

Attachment 6

CompuChem
A Division Of
Liberty Analytical Corp.

Quality Assurance Notice

Case # _____ SDG# _____
Receipt Date _____ Matrix _____

In the USEPA Contract Laboratory Program (CLP) "Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration (Document Number SOM01.2),"directions are provided dealing with a temperature blank, termed the USEPA Cooler Temperature Indicator.

If a cooler temperature indicator bottle is not present in the cooler, the laboratory is required to contact the Sample Management Office (SMO), inform them of that fact and use an alternative means of determining the cooler temperature.

The following is a list of options employed by CompuChem to determine the cooler temperature. For the Case/ SDG presented above, the option(s) used have been indicated by a check mark.

Note: Any of the options performed are done so immediately after the cooler has been opened and the determination made that the cooler temperature indicator bottle is absent.

Water Samples
___An aliquot from a sample bottle designated for extractable organics is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after a 3-minute equilibration period. The contents of the disposable container are then properly discarded.

___A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

Soil Samples
___A calibrated IR gun is used, as indicated for water samples.

As required by the organic SOW, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative.

Signature_____
Date_____

QAN-R-5
011324

Qanr5 – 06/22/10:bm

Attachment 7

CompuChem
A Division Of
Liberty Analytical Corp.

Quality Assurance Notice

Case # _____SDG# _____
Receipt Date _____Matrix _____

In the USEPA Contract Laboratory Program (CLP) "Statement of Work for Inorganics Analysis, Multi-Media, Multi-Concentration (Document Number ILM05.4),"directions are provided dealing with a temperature blank, termed the USEPA Cooler Temperature Indicator.

If a cooler temperature indicator bottle is not present in the cooler, the laboratory is required to contact the Sample Management Office (SMO), inform them of that fact and use an alternative means of determining the cooler temperature.

The following is a list of options employed by CompuChem to determine the cooler temperature. For the Case/ SDG presented above, the option(s) used have been indicated by a check mark.

Note: Any of the options performed are done so immediately after the cooler has been opened and the determination made that the cooler temperature indicator bottle is absent.

Water Samples
___ An aliquot from a sample bottle is poured into a disposable container, a thermometer is inserted into the disposable container, and the temperature is taken and recorded after a 3-minute equilibration period. The contents of the disposable container are then properly discarded.

___ A calibrated IR temperature gun is focused onto a sample container, contained in the cooler, and after a minimum of 5 seconds, a temperature reading is taken and recorded.

Soil Samples
___ A calibrated IR gun is used, as indicated for water samples.

As required by the inorganic SOW, the alternative technique used to determine the cooler temperature must be documented in the SDG Narrative.

Signature_____
Date_____

QAN-R-6
0511159

Qanr6 – 06/22/10:bm

Attachment 8

CompuChem a division of Liberty Analytical Corp. Logbook 7 SS 1

IR Gun Check

The tolerance for the IR Gun check is ± 0.5°C. If this criterion is not met, the IR Gun must be removed for recalibration.
IR calibration is performed daily.

Date	IR Gun No.	Thermometer Temp. °C	Container Type	Container Temp °C	Checked By	Comments
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			
			Clear Glass			
			Amber Glass			
			Plastic			

Reviewed By: _____ Date: _____

08/10/09:bm

Attachment 9

Sample Receiving Walk-in Refrigerator #1 Temperature Log

CompuChem, a division of Liberty Analytical — Logbook 7 A 7

Acceptable temperature range is 2 °C to 6 °C. Record temperatures to the nearest degree. If temperature is not within acceptance range, notify supervisor or maintenance immediately. Temperature readings should be taken at approximately the same time each day. Please indicate if the time recorded is am or pm.

Daily Temperature Reading						Temperature Corrective Action	Mercury Separation		
Date	Time	Temperature (°C)	Initials	AC	UN	If unacceptable, action taken*	Yes	No	If yes, date rejoined** or new thermometer ID

AC = Acceptable UN = Unacceptable

*Once corrective action is completed, document an additional temperature reading to verify unit is within acceptance criteria.

** If mercury separation in the thermometer is evident, notify supervisor immediately so that the mercury can be rejoined following QA SOP 17.12.

Reviewed by: _____ Date: _____ 11/25/08: bm

Note: This page subject to change without notice.

Attachment 10

CompuChem
a division of Liberty Analytical Corp.

CHAIN OF CUSTODY

501 Madison Ave.
Cary, NC 27513
Phone: 919-379-4100 Fax 919-379-4040

Page ____ of ____

Courier
Airbill No.
Sampling Complete? Y or N

Client/Reporting Information	Project Information
Company Name	Project Name
Address	Sampling Location
City State Zip	Turnaround time
Project Contact	Batch QC or Project Specific? If Specific, which Sample ID?
Phone #	Are aqueous samples field filtered for metals? Y or N
Sampler's Name	Are high concentrations expected? Y or N? If yes, which ID(s)?

Requested Analysis (include method and bottle type)

Matrices
GW - Ground water
WW - Waste water
SW - Surface water
SO - Soil/Sediment
TB - Trip Blank
RI - Rinsate
WP - Wipe
O - Other

CompuChem No (Lab Use)	Field ID	Collection Date	Collection Time	Matrix	# of bottles	HCl	NaOH	NHO3	H2SO4	MEOH	Other	pH / Sample Info (Lab Use)

Lab Use Only		Comments
Sample Unpacked By:	Cyanide samples checked for sulfide & chlorine? Y or NA	
Sample Order Entry By:	625 & Phenol samples checked for chlorine? Y or NA	
Samples Received in Good Condition? Y or N	608 samples checked for pH between 5.0-9.0? Y or NA	
If no, explain:		

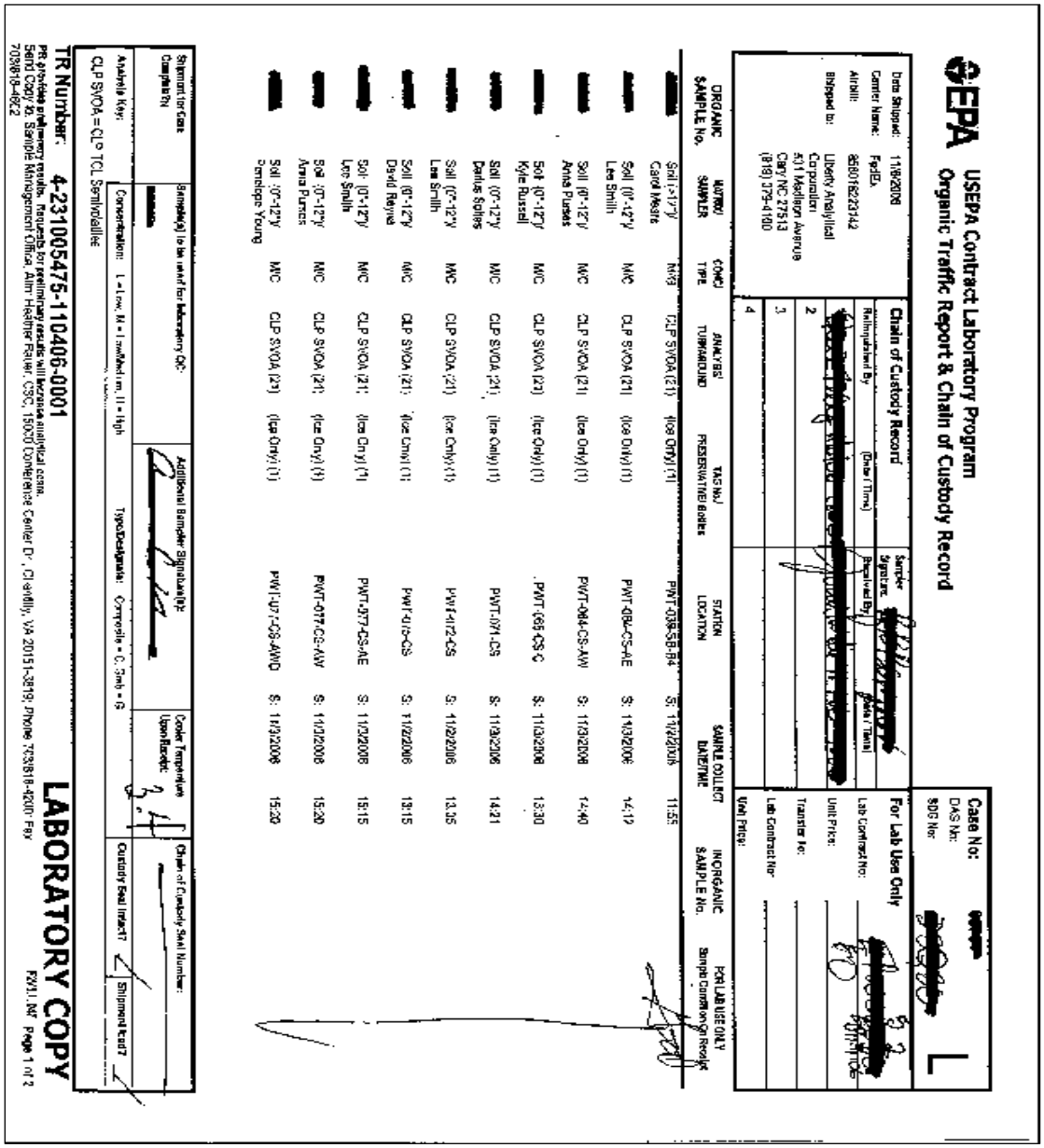
Sample Custody			
Relinquished by:	Date/Time:	Received by:	Date/Time:
Relinquished by:	Date/Time:	Received by:	Date/Time:
Subcontact? Y or N If yes, where?	Custody Seal(s) intact? Y or N	On Ice? Y or N	Cooler Temp: °C

Samples stored 60 days after date report mailed at no extra charge.

White & Yellow copy to lab • Pink copy for customer

Note: This page subject to change without notice.

Attachment 11



EPA USEPA Contract Laboratory Program
Organic Traffic Report & Chain of Custody Record

Date Shipped: 11/8/2008
Carrier Name: FedEx
Airbill: 8580162231 42
Shipped to: Liberty Analytical Corporation 501 Madison Avenue Cary NC 27513 (919) 379-4100

Chain of Custody Record
Relinquished By (Date / Time) [redacted]
Received By (Date / Time) [redacted]
Sampler Signature: [redacted]

Case No: [redacted]
DAS No:
SDG No: [redacted]
L

For Lab Use Only
Lab Contract No:
Unit Price:
Transfer No:
Lab Contract No:
Void Price:

ORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS/ TURNAROUND	TAG No./ PRESERVATIVES Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	INORGANIC SAMPLE No.	FOR LAB USE ONLY Sample Condition On Receipt
[redacted]	Soil (0"-12") Carol Meade	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-039-SB-B4	11/8/2008 11:55		
[redacted]	Soil (0"-12") Lee Smith	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-009-CS-AE	11/3/2008 14:12		
[redacted]	Soil (0"-12") Anna Pursell	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-044-CS-AW	11/3/2008 14:40		
[redacted]	Soil (0"-12") Kyle Russell	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-068-CS-C	11/3/2008 13:30		
[redacted]	Soil (0"-12") Dallas Collins	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-071-CS	11/3/2008 14:21		
[redacted]	Soil (0"-12") Lee Smith	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-072-CS	11/2/2008 13:35		
[redacted]	Soil (0"-12") David Rayner	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-076-CS	11/2/2008 13:15		
[redacted]	Soil (0"-12") Lee Smith	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-077-CS-AE	11/2/2008 13:15		
[redacted]	Soil (0"-12") Anna Pursell	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-077-CS-AW	11/2/2008 13:30		
[redacted]	Soil (0"-12") Penelope Young	MVC	CLP SVOA (21)	(Ice Only) (1)	PWT-077-CS-AWD	11/2/2008 13:30		

Shipment for Case: Complete Y/N
Sample(s) to be used for Laboratory QC: [redacted]
Additional Sampler Signature(s): [redacted]
Cooler Temperature Upon Receipt: 3.4
Chain of Custody Seal Number:

Analysis Key: CLP SVOA = CLP TCL Semivolatiles
Concentration: L = Low, M = Low/Medium, H = High
Type/Designate: Composite = C, Grab = G
Custody Seal Intact? Y
Shipment Intact? Y

TR Number: 4-231005475-110406-0001
PR priority preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, Attn: Heather Fauver, CSC, 15000 Conference Center Dr., Chantilly, VA 20151-3819; Phone 703/818-4200; Fax 703/818-4662

LABORATORY COPY
FX11.INF Page 1 of 2

Attachment 12

USEPA Contract Laboratory Program
Inorganic Traffic Report & Chain of Custody Record

Case No: [redacted]
CAS No:
SDG No: [redacted]
L

Date Shipped: 11/7/2008
Carrier Name: FedEx
Airbill: 854215443449
Shipped to: Liberty Analytical Corporation, 501 Madison Avenue, Cary NC 27513, (919) 379-4100

Chain of Custody Record

Relinquished By	(Date / Time)	Received By	(Date / Time)
[redacted]		[redacted]	
2			
3			
4			

Sampler Signature: [redacted]

For Lab Use Only
Lab Contract No: [redacted]
Unit Price: NA
Transfer To:
Lab Contract No:
Unit Price:

INORGANIC SAMPLE No.	MATRIX/ SAMPLER	CONC/ TYPE	ANALYSIS TURNAROUND	TAG No./ PRESERVATIVE/ Bottles	STATION LOCATION	SAMPLE COLLECT DATE/TIME	ORGANIC SAMPLE No.	FOR LAB USE ONLY Sample Condition On Receipt
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	118 (Ice Only) (1)	SS49	S: 11/7/2008 14:30		GOOP
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	119 (Ice Only) (1)	SS50	S: 10/31/2008 15:20		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	120 (Ice Only) (1)	SS51	S: 10/31/2008 16:00		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	121 (Ice Only) (1)	SS52	S: 11/6/2008 15:00		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	122 (Ice Only) (1)	SS53	S: 11/7/2008 9:45		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	123 (Ice Only) (1)	SS54	S: 11/7/2008 10:00		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	124 (Ice Only) (1)	SS55	S: 11/7/2008 10:15		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	125 (Ice Only) (1)	SS56	S: 11/7/2008 11:15		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	126 (Ice Only) (1)	SDSP	S: 11/6/2008 12:30		
[redacted]	Soil/Sediment/ Michelle Dallessandro	L/G	As, Pb (14)	127 (Ice Only) (1)	SDUP	S: 11/6/2008 12:45	Site-Local Sample	

Shipment for Case Complete Y: Sample(s) to be used for laboratory QC: [redacted]
Additional Sampler Signature(s):
Cooler Temperature Upon Receipt: 2.8
Chain of Custody Seal Number:
Analysis Key: As, Pb = As, Pb
Concentration: L = Low, M = Low/Medium, H = High
Type/Designate: Composite = C, Grab = G
Custody Seal Intact? ✓ Shipment Iced? ✓

TR Number: 3-532217917-110706-0001

LABORATORY COPY

PR provides preliminary results. Requests for preliminary results will increase analytical costs.
Send Copy to: Sample Management Office, 2000 Edmund Halley Dr., Reston, VA 20191-3400 Phone 703/264-9346 Fax 703/264-9222

P2V8.1.043 Page 1 of 1

Attachment 13

Page ___of ___

CompuChem, a Division of Liberty Analytical Corp.- EPA ORGANIC RECEIVING LOG

Client / Account EPA	Rec'd Date	Comments:
Case No:	Courier	
Temp Blk in cooler? Yes / No	Airbill No.	
Temperature:		
Tags? Yes / No		
Custody Seals? Yes / No Intact? Yes / No		

COOLER REC'D BY:	REVIEWED BY:
Samples Logged in by:	SDG #:
Number of TR's?	Work Order No.:
Return Airbill in cooler? Yes / No	

No.	Sample No.	Client ID	QC	Matrix	Sample Date	Sample Time	T. VOC	VOC	VOC SIM	SVOC	SIM	PEST	AR.	Amount/Containers
1														
2														
3														
4														
5														
6														
7														
8														
9														
10														
11														
12														
13														
14														
15														
16														
17														
18														
19														
20														

OrgRec.log.doc-02/24/06

Attachment 14

Page ___of

CompuChem, a Division of Liberty Analytical Corp.- EPA INORGANIC RECEIVING LOG

Client / Account EPA	Rec'd Date	Comments:
Case No:	Courier	
Temp. Blk in cooler? Yes / No	Airbill No.	
Temperature:		
Tags? Yes / No		
Custody Seals? Yes / No Intact? Yes / No		

COOLER REC'D BY:	REVIEWED BY:
SAMPLE LOGIN:	SDG #:
Number of TR's?	Work Order No:
Return Airbill in Cooler? Yes / No	
Cyanide Samples checked for sulfide & chlorine? YES / NA	

No.	Sample No.	Client ID	QC	Matrix	Sample Date	Sample Time	Total Metals	Cyanide	Dissolved Metals	pH	Amount/Containers
1											
2											
3											
4											
5											
6											
7											
8											
9											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											

InorgReclog.doc-03/01/06

Attachment 15

SAMPLE LOG-IN SHEET
FORM DC-1

Lab Name:	Page of
Received By (Print Name)	Log-in Date
Received By (Signature)	
Case Number	Sample Delivery Group No. / Mod. Ref. No.

Remarks:

1. Custody Seal(s) — Present/Absent* Intact/Broken
2. Custody Seal Nos. _____ _____
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists — Present/Absent*
4. Airbill — Airbill/Sticker Present/Absent*
5. Airbill No. _____ _____
6. Sample Tags* — Present/Absent*
   Sample Tag Numbers — Listed/Not Listed on Chain-of-Custody
7. Sample Condition — Intact/Broken*/ Leaking
8. Cooler Temperature Indicator Bottle — Present/Absent*
9. Cooler Temperture ____
10. Does Information on TR/COCs and sample tags agree? — Yes/No*
11. Date Received at Lab _____
12. Time Received _____

EPA Sample #	Corresponding Sample Tag #	Corresponding Assigned Lab#	Remarks: Condition of Sample Shipment, etc

Sample Transfer	
Fraction	Fraction
Area #	Area #
By	By
On	On

* Contact SMO and attach record of resolution

Reviewed By		Logbook No.
Date		Logbook Page No.

SOM01.2 (8/2007)

Attachment 15 (continued)

SAMPLE LOG-IN SHEET

Lab Name:	Page of
Received By (Print Name)	Log-in Date
Received By (Signature)	
Case Number	Sample Delivery Group No. / Mod. Ref. No.

Remarks:

1. Custody Seal(s) — Present/Absent* Intact/Broken
2. Custody Seal Nos. _____ _____
3. Traffic Reports/ Chain of Custody Records (TR/COCs) or Packing Lists — Present/Absent*
4. Airbill — Airbill/Sticker Present/Absent*
5. Airbill No. _____ _____
6. Sample Tags* — Present/Absent*
   Sample Tag Numbers — Listed/Not Listed on Chain-of-Custody
7. Sample Condition — Intact/Broken*/ Leaking
8. Cooler Temperature Indicator Bottle — Present/Absent*
9. Cooler Temperture ____
10. Does Information on TR/COCs and sample tags agree? — Yes/No*
11. Date Received at Lab _____
12. Time Received _____

EPA Sample #	Aqueous sample pH	Corresponding Sample Tag #	Corresponding Assigned Lab#	Remarks: Condition of Sample Shipment, etc

Sample Transfer

Fraction	Fraction
Area #	Area #
By	By
On	On

* Contact SMO and attach record of resolution

Reviewed By		Logbook No.
Date		Logbook Page No.

FORM DC-1 ILM05.4

Attachment 16

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

A chlorine and sulfide check was performed on the above cyanide sample

The results are checked below.

Chlorine was detected _____
Sulfide was detected _____

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

1. Client notified laboratory that data **would** be reported to the State of North Carolina. _____

2. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Project Manager/ID _____/_____ Date _____

QAN-R-7
060126

Qanr7 – 1/26/06:jad

Attachment 16 (continued)

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis _____
Receipt Date _____

The pH reading for the sample above was _____: the required pH level is _____.

A CompuChem Project Manager contacted the client who instructed the laboratory to:

Preserve in-house _____

Note: For samples preserved in house, certain clients require that the maximum amount of preservative added to a sample in an SDG also be added to the associated field or equipment blank. If neither blank is present, the appropriate laboratory must be notified so the proper amount of preservation can be added to the method blank.

Analyze - qualify with notice _____

3. Client notified laboratory that data **would** be reported to the State of North Carolina. _____

4. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Subcontract lab to preserve _____

Project Manager_____ Date _____

Preservation Type_____ Preservative Lot Number_____

Preserved By_____ Date_____

QAN-R-8
060126

QAN-R-8:012606:jad

Attachment 16 (continued)

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

Client _____
Case # _____
Type of Analysis _____
Receipt Date _____

For some organic and/or inorganic determinations temperature preservation at 4° C is required for environmental samples during shipment to the laboratory and prior to analysis. A temperature tolerance range is generally allowed. Temperature of a representative sample from the shipping container is taken and recorded by the receiving clerk at the time of sample receipt. This temperature is representative of all samples contained in the cooler. The EPA CLP program requires the laboratory make notification when the temperature exceeds 10° C. The State of North Carolina requires that samples must be iced to above freezing but ≤ 6° C during shipment. Notification to other clients is either client or project dependent.

Samples that are hand delivered to the laboratory immediately after collection may not meet this criteria. In these cases, the samples shall be considered acceptable if there is evidence that the chilling process has begun, such as arrival on ice.

The temperature of this sample at the time of receipt was determined to be _____.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Hand Delivery/Received on ice _____

Analyze - qualify with notice _____

1. Client notified laboratory that data **would** be reported to the State of North Carolina. _____

2. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Project Manager/ID _____/_____ Date _____

QAN-R-9
060309

qanr9 – 3/09/06:jad

Attachment 16 (continued)

CompuChem
A Division Of
Liberty Analytical Corp.

QUALITY ASSURANCE NOTICE
(North Carolina Samples Only)

CompuChem ID # _____
Client ID # _____
Case # _____
Type of Analysis: Sulfide in Water___
Receipt Date _____

The above water sample was submitted for sulfide analysis.

At the time of sample receipt, headspace was observed in the sample.

A CompuChem customer service representative contacted the client. The client instructed the Receiving department to:

Analyze - qualify with notice _____

1. Client notified laboratory that data **would** be reported to the State of North Carolina. _____
2. Client notified laboratory that data **would not** be reported to the State of North Carolina. _____

Dispose - client will resample _____

Project Manager/ID _____/_____ Date _____

QAN-R-10
090810

Qanr10 – 08/10/09:bm

Attachment 17

CompuChem, a Division of Liberty Analytical Corp. Logbook 7 FF 8

Sample Receipt/Storage Log

Date Received	SDG	Labeled By	Stored By

Reviewed By: _____ Date: _____

7/30/08:vr

SOP 12.1

CompuChem
A Division Of
Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513

NELAP accredited

SOP DOCUMENTATION FORM

This form must accompany all new and revised Standard Operating Procedures (SOPs) when you turn them in to Quality Assurance for review. Please fill out the entire block below (except effective date).

This is a new procedure _____ revised procedure _____ outdated procedure (archive) _____

- Procedure Code: _____ SOP Section #: 12.1 Revision #: 8

SOP Title: Hazardous Waste Disposal

Effective date: (QA fills in) 08/18/2010

- Procedure prepared by: Judith Dozier Date: 08/18/2010
- Procedure approved by: (If the manager prepared the SOP, a qualified second party should sign) [illegible] Date: 8-18-2010
- Reason for change: Update References
- This procedure meets the requirements of the following approved method references: RCRA 40 CFR (261-271); OSHA 29 CFR (1910.120 & 1910.1200); HMTA 40 CFR (171-180: HM-181 & HM126F); CWA 40 CFR (403.5); SARA 40 CFR (355,370); 15A NCAC 13A; Dept of Ag. 7 CFR (301.81)

Procedure approved by a QA Representative (unless QA Representative signed above):

_____ Date: _____

On an annual basis: Lab managers are required to review lab practices and revise the SOP if necessary. If no revision is necessary, indicate by your signature that the SOP has been reviewed.

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Annual Review—Signature: _____ Date: _____

Hazardous Waste Management **and** Safety SOP 12.1: Hazardous Waste Disposal

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ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

1.0 Scope and Application

This SOP provides guidelines for the safe and legal collection, storage, and disposal of all wastes generated by the laboratory's operations. These guidelines are applicable to all employees and subcontractors employed to handle samples, standards, or laboratory chemicals.

Staff members performing the procedures described in this SOP are responsible for reading, understanding, and complying with the SOP requirements. Supervisors are responsible for directing the analyst to the controlled SOP, and providing adequate explanation of the material contained therein.

This procedure is restricted to use by or under the supervision of employees experienced or trained in the processes described below.

2.0 Summary

All samples received and all waste generated by the laboratory in sample processing must be handled and disposed of in a manner that is compliant with state and federal regulations. Procedures are outlined for the safe handling and disposition of waste comprising a variety of classifications or “streams”. Staff who manage the hazardous waste are identified and receive specific on-going training.

3.0 Definitions

3.1 Hazardous Waste Technician - The hazardous waste technician is responsible for the following duties:

3.1.1 Handles all laboratory wastes streams as described in **Section** 7.0 of this SOP.

3.1.2 Interacts with waste disposal companies to schedule waste removal.

3.1.3 Studies applicable ways to reduce the amount of hazardous waste generated.

3.1.4 Trains other employees in specific hazardous waste management when necessary.

3.1.5 Serves as an emergency coordinator.

3.2 Chemical Hygiene Officer - The Chemical Hygiene Officer is responsible for the following duties:

3.2.1 Coordinates training sessions and information exchange concerning chemical spills, waste handling, and the use of emergency equipment.

3.2.2 Investigates and reports accidents.

3.2.3 Develops emergency plans and spill response.

3.2.4 Reviews waste handling and disposal procedures.

3.2.5 Serves as an emergency coordinator.

3.3 Sample Custodians

3.3.1 The Sample Custodians dispose of expired samples in the appropriate drum in their satellite area.

3.4 Laboratory Supervisors

3.4.1 Laboratory Supervisors are responsible for implementing these guidelines.

3.5 Laboratory Staff

3.5.1 Laboratory Staff are responsible for following these guidelines. Waste minimization efforts by laboratory staff are also encouraged.

3.6 Emergency Coordinators

3.6.1 Detailed responsibilities of Emergency Coordinators are outlined in the CompuChem Contingency Plan.

3.7 Waste Minimization Coordinator

3.7.1 The Waste Minimization Coordinator maintains and oversees the company's Waste Minimization Plan. The Hazardous Waste Technician currently handles the responsibilities of this position.

3.8 Waste Streams - The following list describes each type of waste stream generated at CompuChem.

3.8.1 Waste Methylene Chloride

3.8.1.1 Used as a solvent in various extractions and to clean glassware

3.8.2 Waste Freon 113

3.8.2.1 Used in Total Petroleum Hydrocarbon extractions

3.8.3 Waste Mixed Flammable Solvents

3.8.3.1 Used in extractions

3.8.4 Plants Scraps

3.8.4.1 Solid wastes, such as filters, left from laboratory processes

3.8.5 Flammable Solvents in Vials

3.8.5.1 Very small or unopenable vials produced by GC, GC/MS, and other labs

3.8.6 019 Waste

3.8.6.1 Acidic extraction waters, purged water samples, and waste mixed acids that don't meet RCRA metal regulations, contain solvents or are otherwise contaminated, and are ineligible for disposal to the sewer system

3.8.7 Waste Mixed Acids

3.8.7.1 Cyanide distillations, ICP, AA instruments, digestates, and glassware acid bath

3.8.8 Waste Sodium Hydroxide and Cyanide

3.8.8.1 Cyanide distillations

3.8.9 Purged Soil Samples

3.8.9.1 Expired solid samples from the sample cooler

3.8.10 Empty Sample Containers

3.8.10.1 A non-hazardous waste stream from the purged water samples and other sources

3.8.11 Broken Glassware

3.8.11.1 A non-hazardous waste stream that comes from all laboratory processes and glassware prep.

3.8.12 Expired Chemicals

3.8.12.1 Expired reagents and standards from all labs

3.8.13 Purged Water Samples

3.8.13.1 Expired water samples from the sample cooler

3.8.14 Other

3.8.14.1 The Chemical Hygiene Officer and the Hazardous Waste Technician will specify other waste streams as needed.

3.9 Waste Disposal Companies

3.9.1 To prevent improper disposal, the licensed companies that dispose of this laboratory's hazardous waste perform additional testing before disposal to determine the contents of the shipment containers.

3.10 RCRA – Resource Conservation and Recovery Act

3.11 DOT – Department of Transportation

4.0 <u>Safety</u>

4.1 Emergency Procedures

4.1.1 Contingency Plan

4.1.1.1 All emergency procedures may be found in the facility's Contingency Plan.

4.2 Appropriate protective equipment and clothing must be used under the assumption that all samples are potentially hazardous. Safety glasses, gloves and lab coats are a minimum requirement. The persistent presence of noxious odors may be indicative of failure of the laboratory ventilation system and must be reported to a supervisor or manager.

4.3 Laboratory staff **must** review the Chemical Hygiene Plan for general safety policies, and Material Safety Data Sheets for reagents used in the laboratory.

5.0 Equipment **and** Supplies

5.1 Moving Equipment

5.1.1 The solvent storage/waste staging room is stocked with equipment, such as handtrucks and a drum cart, to help in the movement of containers of waste.

5.2 Waste Compactor

5.2.1 An electric compactor is located outside and is used to compact the purged soil sample and empty sample container waste streams.

6.0 Sample Preservation **and** Storage

N/A

7.0 Quality Control

7.1 Disposal Arrangements

7.1.1 All waste is handled in compliance with all applicable local, state, and federal laws. A licensed waste disposal company (or companies) is contracted to dispose of hazardous wastes.

7.2 Compliance with Federal Regulations

7.2.1 The laboratory is required to comply with applicable parts of the regulations listed in Section 11.0 of this SOP.

7.3 All personnel receive training by the Human Resources department covering "Right to Know", the Chemical Hygiene Plan, OSHA Standards, and the Contingency Plan/Emergency Action Plan.

7.4 Employees identified as hazardous waste handlers or managers receive additional, more specific training including hazardous waste management, RCRA and DOT regulations, and managing hazardous waste and used oil, for example. Continued training is required to be received on an annual basis and may be administered internally by trained laboratory staff or through an external provider. An example of the training checklist used for internal training is provided as Attachment 1. Certificates and other documentation are maintained in the employee training files.

7.5 Logbook Review

7.5.1 All logbooks are reviewed by the manager and audited on a periodic basis by the Quality Assurance department.

8.0 Calibration **and** Standardization

N/A

9.0 Procedure

9.1 Documentation Requirements

Documentation must follow the requirements in QC SOP **13.6**: **"**Proper Documentation Procedure**"**. Documentation requirements include the following.

9.1.1 Hazardous Waste Manifest

9.1.1.1 A North Carolina Hazardous Waste Manifest (Attachment 2) is completed and accompanies all shipments to the licensed hazardous waste contractor. The manifest is signed by the generator of the hazardous waste and the transporter to acknowledge receipt of the materials. When the materials have been disposed of, a completed copy of the manifest with signatures from the disposal facility is returned to the laboratory. The Hazardous Waste Technician maintains a file of these records.

9.1.2 Sample Accident Report (Attachment 3) and Spill Report (Attachment 4)

9.1.2.1 The Sample Accident Report and Spill Report must be completed for each event and are administered by the Chemical Hygiene Officer.

9.1.2.2 Spills are handled according to the guidelines in Hazardous Waste Management and Safety SOP 12.2, "Spill Control and Cleanup".

9.1.3 Inspection Logs

9.1.3.1 The following logs must be maintained:

9.1.3.1.1 Outside 90-day Storage Area Daily Inspection. Logbook 18D (Attachment 5).

9.1.3.1.2 Inside 90-day Storage Area Daily Inspection Logbook.

9.1.3.1.3 Manifest Tracking Logbook 18E (Attachment 6).

9.2 Disposal by Waste Stream

The procedure for handling each kind of waste stream is described below. Grounding procedures are described and diagrammed in Attachment 7 and Attachment 8.

9.2.1 Waste Freon 113

9.2.1.1 Disposal Container

9.2.1.1.1 Use a DOT-approved steel 55-gallon closed-head drum for shipment.

9.2.1.2 Personal Protective Equipment

9.2.1.2.1 Wear a lab coat, full-face respirator (or half-face respirator with safety glasses or face shield) and appropriate gloves.

9.2.1.3 Method

9.2.1.3.1 Transfer the Freon 113 into the satellite container by either pouring or pumping. Fill to within about two inches of the top of the drum.

9.2.1.4 Storage Location and Labeling

9.2.1.4.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.2 of this SOP. Place a drain mat over any drain en route to the outside storage area.

9.2.2 Waste Mixed Flammable Solvents

9.2.2.1 Special Instructions

9.2.2.1.1 Use grounding method #1 when pouring from mixed flammable solvent satellite containers into the 55-gallon drum.

9.2.2.2 Disposal Container

9.2.2.2.1 Use a DOT-approved steel 55-gallon closed-head drum for shipment.

9.2.2.3 Personal Protective Equipment

9.2.2.3.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be used.

9.2.2.4 Method

9.2.2.4.1 Transfer the solvents from the satellite container into the drum by pouring. Fill to within about two inches of the top of the drum.

9.2.2.5 Storage Location and Labeling

9.2.2.5.1 The filled container should be taken to the outside storage area and be labeled as directed in Section 9.3.3 of this SOP. Place a drain mat over any drain enroute to the outside storage area.

9.2.3 Plant Scraps

9.2.3.1 Disposal Container

9.2.3.2.1 Use a DOT-approved 55-gallon open-head steel drum or plastic drum.

9.2.3.2 Personal Protective Equipment

9.2.3.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be used.

9.2.3.3 Method

9.2.3.3.1 Transfer the plant scraps from the satellite container into the steel drum. Use the inner liner of a satellite container or some other flat object to compact the plant scraps so that as much waste as possible may be placed in a container. Compacting is optional.

9.2.3.4 Storage Location and Labeling

9.2.3.4.1 Filled containers should be taken to the outside storage area and be labeled as directed in Section 9.3.4 of this SOP.

9.2.4 Flammable Solvent in Vials

9.2.4.1 Disposal Container

9.2.4.1.1 Transfer the vials from the satellite container into a DOT-approved 55-gallon open-head steel drum.

9.2.4.2 Personal Protective Equipment

9.2.4.2.1 Wear a lab coat, safety glasses, and appropriate gloves.

9.2.4.3 Storage Location and Labeling

9.2.4.3.1 Filled containers should remain in the solvent storage/waste staging room and be labeled as directed in Section 9.3.5 of this SOP.

9.2.5 019 Waste

9.2.5.1 Disposal and Storage Containers

9.2.5.1.1 Transfer 019 waste from satellite containers into a DOT-approved steel 55-gallon closed-head drum or plastic drum.

9.2.5.2 Purged Water Samples

9.2.5.2.1 Transfer liquid samples from cooler. Empty the sample bottles by purging directly into a DOT-approved 55-gallon closed-head drum. Dispose of the empty sample container according to section 9.2.10.

9.2.5.3 Personal Protective Equipment

9.2.5.3.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be used.

9.2.5.4 Storage Location and Labeling

9.2.5.4.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.6 of this SOP. Place a drain mat over any drain en route to the outside storage area.

9.2.6 Waste Mixed Acids

9.2.6.1 Storage Containers

9.2.6.1.1 Transfer waste mixed acids from satellite containers into a 55-gallon non-metal, acid-resistant drum for storage.

9.2.6.2 Personal Protective Equipment

9.2.6.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be worn.

9.2.6.3 Storage Location and Labeling

9.2.6.3.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.7 of this SOP. Place a drain mat over any drain en route to the outside storage area.

9.2.7 Waste Sodium Hydroxide and Cyanide

9.2.7.1 Storage Container

9.2.7.1.1 Transfer the waste sodium hydroxide and cyanide from satellite containers to a 55-gallon non-metal, corrosion-resistant drum for storage.

9.2.7.2 Personal Protective Equipment

9.2.7.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A respirator may also be worn.

9.2.7.3 Storage Location and Labeling

9.2.7.3.1 Filled containers should be taken to the outside storage area and labeled as directed in Section 9.3.8 of this SOP. Place a drain mat over any drain en route to the outside storage area.

9.2.8 Purged Soil Samples

9.2.8.1 Disposal Container

9.2.8.1.1 Place purged soil samples with containers into a DOT-approved 55-gallon, open-head steel drum.

Note: The identifying labels, including client or project names and other information, must be removed prior to disposal.

9.2.8.2 Personal Protective Equipment

9.2.8.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A face shield may also be worn.

9.2.8.3 Storage Location and Labeling

9.2.8.3.1 Filled containers should be moved to the outside storage area and be labeled as directed in Section 9.3.9 of this SOP.

9.2.8.4 Foreign Soil and Domestic Quarantine Soil Samples

9.2.8.4.1 All samples arriving from outside the continental United States and from quarantined areas of the continental United States, are to be heated to 500 °F for two minutes prior to being disposed in the manner that domestic soils are disposed. These samples are identified by the presence of red- and white-striped tape on the sample container.

- Option 1: Place the soil samples with container on a metal tray in the muffle furnace in the Sample Preparation Laboratory. Heat to 500°F for two full minutes. Dispose of the cooled samples in accordance with this SOP.

- Option 2: Samples and sample containers may be placed in a 55 gallon, open-head steel drum marked "purged soil, foreign and domestic quarantine". This container can be shipped as is to a waste incineration facility that is permitted by the USDA for foreign and domestic soil disposal.

Note: This includes the filter and residue, if TCLP leaching is done.

9.2.9 Empty Sample Containers

9.2.9.1 Storage Container

9.2.9.1.1 Empty and broken sample containers should have labels removed and are then disposed of in a commercial dumpster.

9.2.9.2 Personal Protective Equipment

9.2.9.2.1 Wear a lab coat, safety glasses, and appropriate gloves. A face shield may also be worn.

9.2.10 Broken Glassware

9.2.10.1 Disposal Container

9.2.10.1.1 Broken glassware will be transferred from the satellite container directly to a dumpster.

9.2.10.2 Personal Protective Equipment

9.2.10.2.1 Wear a lab coat, safety glasses, and appropriate cut-proof gloves (Kevlar, for example). Also wear a pair of flexible gloves over the cut-proof gloves (latex, for example).

9.2.10.3 Method

9.2.10.3.1 Transfer the broken glassware container to the dumpster using the hand trucks. Transfer the container into the dumpster.

9.2.11 Expired Chemicals and Standards

9.2.11.1 When a chemical reaches its shelf life, it must be disposed of as hazardous waste. These chemicals should be boxed and given to the Hazardous Waste Technician for disposal. The hazard class of each chemical should be provided to the Waste Technician with the chemicals so that the chemicals can be directed to the proper waste stream.

9.2.11.2 Hazard Classes and Characteristics

- Non-Halogenated Flammables – Acids
- Halogenated Flammables – Bases
- Pesticides – Poisons
- Oxidizers – Reactives
- Inorganics (dry)

9.2.11.3 Disposal by Hazard Class

9.2.11.3.1 Non-Halogenated Flammable

- Add to the mixed flammable solvent waste stream as described above.

9.2.11.3.2 Halogenated Flammables

- Must be lab-packed or bulked in a 5-gallon DOT-approved steel pail.

9.2.11.3.3 Pesticides

- Must be lab-packed

9.2.11.3.4 Oxidizers

- Must be stored away from organic material

9.2.11.3.5 Inorganic Chemicals (dry)

- Stable chemicals can be lab-packed for incineration.

9.2.11.3.6 Acids

- If clean, acids can be directed to the waste mixed acid stream for disposal to the sewer as described above. Otherwise, they should be directed to the 019 waste stream.

9.2.11.3.7 Bases

- If clean, bases can be used to neutralize clean acid waste. Otherwise, they should be lab-packed for disposal by incineration.

9.2.11.3.8 Poisons

- Will be assessed for other characteristics and directed to the appropriate waste stream

9.2.11.3.9 Reactives

- If the chemical will react with water or air, or will produce a toxic gas when mixed with another chemical, the material should be lab-packed for disposal by incineration.

9.2.12 Purged Water Samples

9.2.12.1 Disposal Container

9.2.12.1.1 For amber liters, gallons and plastics, see Section 9.2.9.

9.2.12.1.2 For volatile samples, the container and sample are placed in a 55-gallon drum marked "vials **and** acid."

9.2.12.2 Personal Protective Equipment

9.2.12.2.1 Wear a lab coat, safety glasses, and appropriate gloves.

9.2.12.3 Method

9.2.12.3.1 The Sample Custodian is responsible for providing water samples to the Hazardous Waste Technician for disposal. The samples are bulked into the above-described container. Only clear samples are to be bulked. Unclear samples are to be diverted to the 019 water waste stream.

9.2.12.4 Storage Location and Labeling

9.2.12.4.1 Filled containers should be moved to the outside storage area and be labeled.

9.2.12.4.2 See Section 9.2.6.3 for Waste Mixed Acids.

9.2.13 Other Wastes

9.2.13.1 All other wastes will be evaluated by the Safety Officer, the Hazardous Waste Technician, and if necessary by the contracted disposal company for the appropriate means of disposal.

9.3 Labeling Requirements for Waste Streams

Examples of labels required for satellite accumulation containers and shipment containers can be found in Attachment 9 and Attachment 10.

9.3.1 Waste Freon 113

9.3.1.1 Satellite Accumulation containers must be labeled with the following information:

9.3.1.1.1 Hazardous Waste Label

9.3.1.1.2 Hazard Index:

- Health: 2
- Flammability: 1
- Reactivity: 0

9.3.1.2 Shipment Containers must be labeled according to 49 CFR 172.101.

9.3.2 Waste Mixed Flammable Solvents

9.3.2.1 Satellite accumulation containers must be labeled with the following information:

9.3.3.1.1 Hazardous Waste Label

9.3.3.1.2 Hazard Index:

- Health: 3
- Flammability: 3
- Reactivity: 0
- Flammable Liquid Label

9.3.2.2 Shipment Containers must be labeled according to 49 CFR 172.101.

9.3.3 Plant Scraps

9.3.3.1 Satellite accumulation containers must be labeled with the following information:

9.3.3.1.1 Hazardous Waste Label

9.3.3.1.2 Hazard Index:

- Health: 4
- Flammability: 3
- Reactivity: 0
- Cancer Hazard Label
- Flammable Liquid Label

9.3.3.2 Shipment containers must be labeled according to 49 CFR 172.101. See Attachment 9 for these requirements.

9.3.4 Flammable Solvent in Vials

9.3.4.1 Satellite accumulation containers must be labeled with the following information:

9.3.4.1.1 Hazardous Waste Label

9.3.4.1.2 Hazard Index:

- Health: 2
- Flammability: 3
- Reactivity: 0
- Flammable Liquids Label

9.3.4.2 Shipment containers must be labeled according to 49 CFR 172.101.

9.3.5 019 Waste

9.3.5.1 Satellite Accumulation containers must be labeled with the following information:

9.3.5.1.1 Hazardous Waste Label with the words "019"

9.3.5.1.2 Hazard Index:

- Health: 3
- Flammability: 2
- Reactivity: 3
- Corrosive Label
- Oxidizer Label

9.3.5.1.3 Sample bottles that contain 019 waste are not required to be labeled.

9.3.5.2 Shipment containers must be labeled according to 49 CFR 172.101.

9.3.6 Waste Mixed Acids and Vials **and** Acid

9.3.6.1 Satellite accumulation containers must be labeled with the following information:

9.3.6.1.1 Hazardous Waste Label

9.3.6.1.2 Hazard Index:

- Health: 3
- Flammability: 0
- Reactivity: 2
- Corrosive Label
- Oxidizer Label

9.3.6.2 Storage Containers must be labeled with the following information:

- Hazardous Waste Label b/w Non-hazardous Waste Label.
- Accumulation Start Date

9.3.7 Waste Sodium Hydroxide with Cyanide

9.3.7.1 Satellite Accumulation containers must be labeled with the following information:

9.3.7.1.1 Hazardous Waste Label

9.3.7.1.2 Hazard Index:

- Health: 3
- Flammability: 0
- Reactivity: 3

9.3.7.1.3 Corrosive Label

9.3.7.2 Storage containers must be labeled with the following information:

9.3.7.2.1 Hazardous Waste Label b/w Non-hazardous waste label

9.3.7.2.2 Accumulation Start Date

9.3.8 Purged Soil Samples

9.3.8.1 Shipment Containers must be labeled according to 49 CFR 172.101.

9.3.9 Empty Sample Containers

9.3.9.1 These Shipment Containers do not have to be labeled. To avoid confusion and inspection, they may be labeled with a "Non-regulated Waste" label. Do not fill in the information on this label.

9.3.10 Broken Glassware

9.3.10.1 The satellite accumulation container does not have to be labeled. To avoid confusion and inspection, it may be labeled with a “Non-regulated Waste” label. Do not fill in the information on this label.

9.3.11 Expired Chemicals

9.3.11.1 All expired chemicals will be lab-packed and, therefore, labels will be used in accordance with the characteristics of the chemicals.

9.3.12 Other Wastes

9.3.12.1 The Chemical Hygiene Officer and/or the Hazardous Waste Technician determine the appropriate labeling requirements.

9.4 Accumulation and Storage Requirements

9.4.1 Satellite Accumulation

9.4.1.1 Time Limitations

9.4.1.1.1 Waste may be accumulated for an indefinite time in satellite accumulation. When the 55 gallon drum is full, it is to be dated and moved to 90 day storage. Once the drum is dated, it must be moved to the 90 day storage within 3 days.

9.4.1.2 Volume Limitations

9.4.1.2.1 No more than 55 gallons of total waste from the same waste stream may be accumulated in satellite containers in one storage area. For example, a laboratory may store flammable liquids in a 55-gallon drum, or it may store many containers whose volume equals 55 gallons.

9.4.1.3 Labeling Limitations

9.4.1.3.1 Hazardous waste labels cannot be dated if they are in satellite accumulation. See Section 9.3 of this SOP for satellite accumulation labeling requirements.

9.4.2 Solvent Storage/Waste Staging Room

9.4.2.1 Time Limitations

9.4.2.1.1 Storage time is limited to 90 calendar days in this room.

9.4.2.2 Labeling Requirements

9.4.2.2.1 Accumulation Start Dates on hazardous waste labels must be filled in once the waste is stored in this room. See Section 9.3 of this SOP for solvent storage/waste staging room requirements.

9.4.3 Outside Storage Area

9.4.3.1 Time Limitations

9.4.3.1.1 Storage time is limited to 90 calendar days in this area.

9.4.3.2 Labeling Requirements

9.4.3.2.1 Accumulation Start Dates on hazardous waste labels must be filled in once the waste is stored in the outside storage area. See also section 9.3 of this SOP for outside storage labeling requirements.

10.0 Waste Management

It is the laboratory's responsibility to comply with all federal, state, and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions, and to protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Compliance with all sewage discharge permits and regulations is also required.

Samples preserved with HCl, HNO_3, NaOH, Zn Acetate, or H_2SO_4 are hazardous and must be neutralized before being disposed, or must be handled as hazardous waste.

Refer to the Hazardous Waste Management and Safety SOPs located in the lab

11.0 References

16.1 Resource Conservation and Recovery Act (RCRA) (40 CFR 261-271)

16.2 Occupational Safety and Health Administration (OSHA) (29 CFR 1910.120 and 1910.1200)

16.3 Hazardous Materials Transportation Act (HMTA) (49 CFR 171-180: HM-181 and HM126F)

16.4 Clean Water Act (CWA) (40 CFR 403.5)

16.5 Superfund Amendments and Reauthorization Act (SARA) (40 CFR 355, 370)

16.6 NC Hazardous Waste Rules (15A NCAC 13A: same as RCRA)

16.7 Department of Agriculture (7CFR 301.81)

16.8 QC SOP **13.6**: **"**Proper Documentation Procedures**"**

16.9 NELAC Standards, **effective July 2003**, plus revisions

16.10 QA-G6: Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations EPA/600/R-96/027, November 1995.

16.11 CompuChem Quality Manual, Revision **13**, **11**/**17**/**09**, plus revisions

16.12 Sample Control SOP 4.1, "Receiving Samples"

16.13 Sample Control SOP 4.6, "Storing Samples"

16.14 Hazardous Waste Management and Safety SOP 12.2, "Spill Control and Cleanup"

12.0 Attachments as Tables, Diagrams, Flowcharts **and** Validation Data

12.1 Attachment 1 – Training Checklist

12.2 Attachment 2 - NC Hazardous Waste Manifest

12.3 Attachment 3 – Sample Accident Report

12.4 Attachment 4 – Spill Report

12.5 Attachment 5 – Outside 90-day Storage Area Daily Inspections Log

12.6 Attachment 6 - Manifest Tracking Log

12.7 Attachment 7 - Schematic of Grounding Method 1

12.8 Attachment 8 - labels for satellite accumulation containers for waste streams

12.9 Attachment 9 – hazardous waste shipment container labels, including a table of DOT-required labels per waste stream

Attachment 1

CompuChem Laboratories

Specific Hazardous Waste Training Record

Employee name: _____ ID no.: _____

Job title: _____

I certify that I have received training in the following areas on the date(s) indicated, and that I understand all information presented.:

Employee Initials	Trainer Initials	Date	Area covered by training
_____	_____	_____	1. Specific hazardous wastes to be managed: _____ _____ _____
_____	_____	_____	2. Proper container(s) to be used
_____	_____	_____	3. Placement of waste into container(s)
_____	_____	_____	4. Required container labels
_____	_____	_____	5. Dating containers
_____	_____	_____	6. Satellite accumulation requirements (if applicable)
_____	_____	_____	7. Acid/base neutralizations
_____	_____	_____	8. Emergency notifications and response
_____	_____	_____	9. Container inspection procedures
_____	_____	_____	10. Lab safety manual hazardous waste policy review

Indicate subject areas in which the employee is not required to be trained by writing an "X" in the date column.

Employee Signature _____ Date _____

Trainer Signature _____ Date _____

Attachment 2

Please print or type. (Form designed for use on elite (12-pitch) typewriter.) Form Approved. OMB No. 2050-0039.

UNIFORM HAZARDOUS WASTE MANIFEST

1. Generator's US EPA ID No. NCR000077160603 — Manifest Document No.
2. Page 1 of 1 — Information in the shaded areas is not required by Federal law.

3. Generator's Name and Mailing Address: CompuChem Environmental Corporation, 501 Madison Ave., Cary NC 27513
4. Generator's Phone (919) 379-4000

A. State Manifest Document Number
B. State Generator's ID

5. Transporter 1 Company Name: T.A.G. Transport — 6. US EPA ID Number: TNR000031111
C. State Transporter's ID
D. Transporter's Phone (865)354-6827

7. Transporter 2 Company Name — 8. US EPA ID Number
E. State Transporter's ID
F. Transporter's Phone

9. Designated Facility Name and Site Address: Perma-Fix of Florida, 1940 NW 67th Place, Gainesville, FL 32653 — 10. US EPA ID Number: FLD980711071
G. State Facility's ID: FLD980711071
H. Facility's Phone (352) 373-6066

11. US DOT Description (Including Proper Shipping Name, Hazard Class and ID Number)	HM	12. Containers No.	Type	13. Total Quantity	14. Unit Wt/Vol	Waste No.
a. Hazardous Waste, Liquid, n.o.s., 9, NA3082, PGIII (contains: methylene chloride, Hexane)	X	009	DF	04320	P	F002/F003
b. Waste Corrosive Liquid,Acidic,Inorganic,N.O.S.,8,UN3264, PGII,(Contains:Hydrochloric Acid,Nitric Acid)	X	001	DF	00398	P	D002
c. RQ,Waste Flammable Liquid,N.O.S.,3,UN1993,PGII (Contains:Methanol,Hexane)	X	003	DM	01350 THT ~~00900~~	P	D001
d. Waste Corrosive Liquid,Acidic,Inorganic,N.O.S.,8,UN3264, PGII,(Contains:Hydrochloric Acid)	X	004	DF	01600	P	D002

J. Additional Descriptions for Materials Listed Above
11a. 019 Water, Profile#29891 D-10 B204 M041
11b. Mixed Acids, Profile#32936-C – D-73 B 104 M141
11c. Mixed Flammables, Profile#32940 – D-73 B204 M061
11d. Vials and Acid, Profile#32937-C D-D610 B104 M141

K. Handling Codes for Wastes Listed Above
S01, M141
D-M141
A-M041 B-M141 -C M061

15. Special Handling Instructions and Additional Information
24 Hour Emergency # 865-376-0193
ERG# 11a.171 11b.154 11c.128 11d.154
RQ=D001>100 lbs

16. **GENERATOR'S CERTIFICATION:** I hereby declare that the contents of this consignment are fully and accurately described above by proper shipping name and are classified, packed, marked, and labeled, and are in all respects in proper condition for transport by highway according to applicable international and national government regulations.
If I am a large quantity generator, I certify that I have a program in place to reduce the volume and toxicity of waste generated to the degree I have determined to be economically practicable and that I have selected the practicable method of treatment, storage, or disposal currently available to me which minimizes the present and future threat to human health and the environment; OR, if I am a small quantity generator, I have made a good faith effort to minimize my waste generation and select the best waste management method that is available to me and that I can afford.

Printed/Typed Name: Carl A. Hooker — Signature: Carl A. Hooker — Month Day Year: 06 08 01

TRANSPORTER

17. Transporter 1 Acknowledgement of Receipt of Materials
Printed/Typed Name: Timothy H. Johnson — Signature: Timothy H. Johnson — Month Day Year: 06 08 01

18. Transporter 2 Acknowledgement of Receipt of Materials
Printed/Typed Name — Signature — Month Day Year

FACILITY

19. Discrepancy Indication Space

20. Facility Owner or Operator: Certification of receipt of hazardous materials covered by this manifest except as noted in Item 19.
Printed/Typed Name: Thomas A. Hamsh — Signature — Month Day Year: 08 13 01

Style CF 17 LABELMASTER, AN AMERICAN LABELMARK CO., CHICAGO, IL 60646 (800)621-5808

EPA Form 8700-22 (Rev. 9-88) Previous editions are obsolete.

ORIGINAL-RETURN TO GENERATOR

PRINTED ON RECYCLED PAPER USING SOYBEAN INK — PRINTED WITH SOY INK

ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 3

CompuChem
a division of Liberty Analytical Corporation

SAMPLE ACCIDENT REPORT

Date & Time accident occurred: _____ / _____

Employee(s) involved: _____

Was anyone injured? ☐Yes ☐No Name: _____

If yes, an injury report must be filled out in the Human Resources Department

Where did accident occur? _____

What analyses were affected? _____

What was the cause of the accident? _____

_____

_____

_____

Sample ID: _____ CCN: _____

Case: _____ SDG: _____

Container size: _____ Bottle ___ of ___

CSR Notified: _____ Date: _____

accform1 – 7/3/01:dce

ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 4

Spill Report

CompuChem a Division of Liberty Analytical Corporation
501 Madison Avenue
Cary, NC 27513

Spill Information			
Date of spill	Time of spill	☐ a. m.	☐ p. m.
Material spilled			
Location of spill			
Brief description of spill:			
Quaintly spilled	Fire? ☐Yes ☐No	Explosion? ☐Yes ☐ No	
Intensity: ☐ Class A fires in ordinary combustible materials (wood, cloth, paper, rubber)			
☐ Class B fires in flammable liquids (oils, greases, tars, flammable gases)			
☐ Class C fires engendered by energized electrical equipment			
☐ Class D fires in combustible metals (Mg, Ti, Zr, Na, Li, K)			
Brief description of fire/explosion:			
Spill contained? ☐ Yes ☐ No	Area evacuated? ☐ Yes ☐ No		
Local authorities contacted? ☐ Yes ☐ No	Emergency response team activated? ☐ Yes ☐ No		
Cleanup procedures used:			
Cleanup personnel:			
Corrective action required to reduce risk:			
Personnel Signature		Date:	
Manager Signature		Date:	
Chemical Hygiene Officer Signature		Date:	

Additional information on back

Attachment 4 (continued)

Personnel Information			
Personnel affected	1.		ID#
	2.		ID#
	3.		ID#
	4,		ID#
Job title 1.		Department: 1.	
2.		2.	
3.		3.	
4.		4.	
Extent of injury			
Describe suspected exposure to hazardous material(s):			
Medical attention required? ☐ Yes ☐ No			
Personnel Signature		Date:	
Manager Signature		Date:	
Chemical Hygiene Officer Signature		Date:	

Attachment 5

COMPUCHEM a division of Liberty Analytical Corp Logbook 18 D 12

Outside 90-Day Storage Area Daily Inspection Log

Date (mm/dd/yy): _____ Inspector: _____

Please check off the following items to verify that each was completed.

☐ Container Condition ☐ Leakage ☐ Bulging ☐ Corrosion ☐ Closed

☐ Lids, caps, & covers on drums

☐ Container properly labeled and dated ☐ Contents indicated on label ☐ "Hazardous Waste" label ☐ Accumulation date on container

☐ Other Container Information ☐ Adequate aisle space present ☐ Contents compatible

☐ Spill kits ☐ Present ☐ Properly labeled

Comments/Corrective Action: _____
_____

Date (mm/dd/yy): _____ Inspector: _____

Please check off the following items to verify that each was completed.

☐ Container Condition ☐ Leakage ☐ Bulging ☐ Corrosion ☐ Closed

☐ Lids, caps, & covers on drums

☐ Container properly labeled and dated ☐ Contents indicated on label ☐ "Hazardous Waste" label ☐ Accumulation date on container

☐ Other Container Information ☐ Adequate aisle space present ☐ Contents compatible

☐ Spill kits ☐ Present ☐ Properly labeled

Comments/Corrective Action: _____
_____

Reviewed by: _____ Date: _____

The presence of the employee's ID number, or signature, on this log attests that strict compliance with the method's SOP has occurred. Any SOP deviations require documentation by the responsible employee together with the employee's initials and the initials of the lab supervisor and a QA department representative, signifying approval of the deviation

9/24/04:jad

ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 6

Manifest Tracking Log

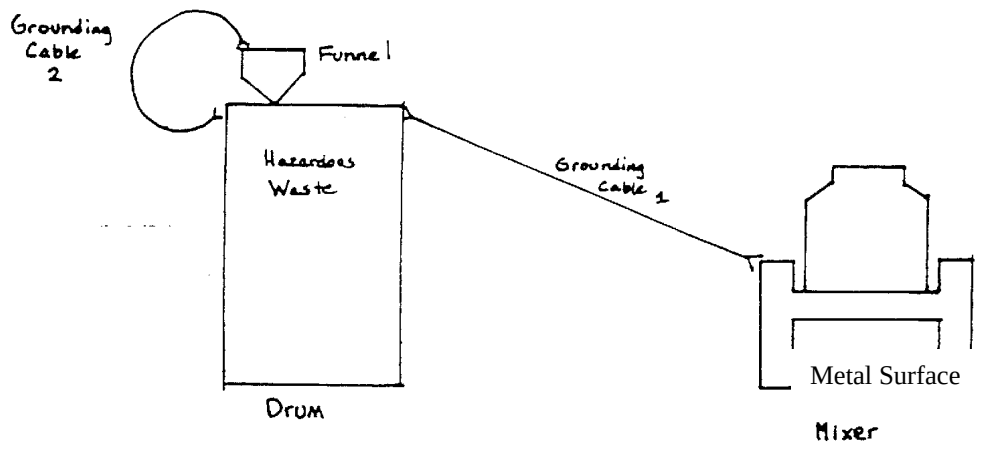
CompuChem a Division of Liberty Analytical Corporation

Number	Waste Shipped	Company	Date Sent	Date Received

Reviewed by: _____ Date: _____

ORIGINAL MASTER COPY CONTROLLED COPY
If words above are not highlighted, this is an uncontrolled copy of this document.

Attachment 7

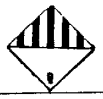
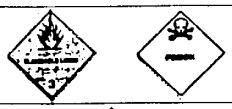
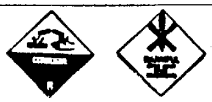
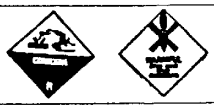


METHOD 1

Fig. 4

Attachment 8

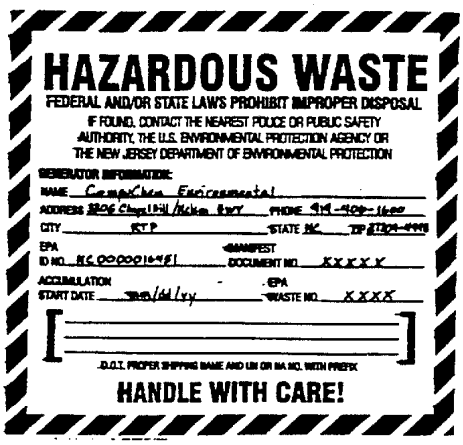
WASTE STREAMS

Profile #	Waste Stream	Code	Shipping Information	RCRA	DOT Label(s)
RVKPU101	Purged Soil Sample	PSSM	Hazardous Waste Solid, NOS (Hexachlorobutadiene, Nitrobenzene) 9, NA 3077, III	F001 F002 F003 F004 F005	
RVKPU102	Flammable Solvent In Vials	SVLS	Waste Flammable Liquid, Poisonous, NOS (Acetone, Methanol) 3, UN 1992, II	D001 F003	
RVKPU103	Plant Scraps	PSWS	Hazardous Waste Solid, NOS (Acetone, Methylene Chloride) 9, NA 3077, III	F002 F003 F004	
RVKPU104	Mixed Flammable Solvents	MFSV	Waste Flammable Liquid, Poisonous, NOS (Methanol, Acetone) 3, UN 1992, II	D001 F003	
RVKPU105	Freon 113	F113	Hazardous Waste Liquid, NOS (1,1,2-Trichlorotrifluoroethane) 9, NA 3082, III	F002	
Left Blank on Purpose					
RVKPU108	019 Water	019W	Hazardous Waste Liquid, NOS (methylene chloride, acetone) 9, NA 3082, III	F002 F003 F005	
RVKPU110	Hardness Reagents	HARD	Ammonium Solution 8, UN 2672, III	D002	
RVKPU111	Ammonia Reagents	AMMO	Waste Corrosive Liquid, NOS (Sodium Hydroxide, Ammonium Chloride) 8, UN 1760, II	D002	
RVKPU112	Phenol Reagents	PHEN	Waste corrosive liquid, poisonous, NOS (Potassium ferrocyanide, boric acid) 8, UN 2922, II	D002	
RVKPU113	Chloride Reagents	CHLR	Waste Corrosive Liquid, Poisonous, NOS (Mercuric thiocyanate, methanol) 8, UN 2922, II	D002	

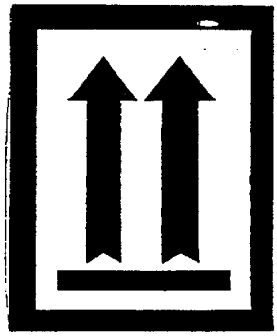
Attachment 9

DOT LABELING SUMMARY

1. Each container of hazardous waste must display a h. label on the top and on one side.



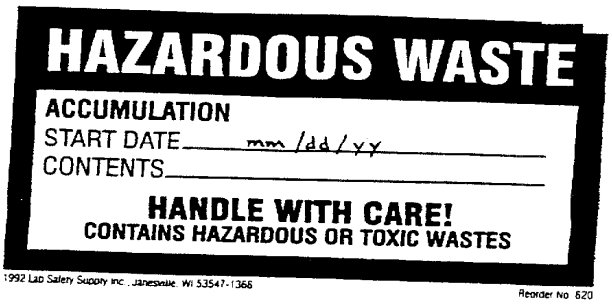
2. Each container of hazardous waste must have two directional labels displayed on opposite sides of the container. When the container is in an upright position, the directional arrows should point upward. Do not affix these labels to the top of the drum!

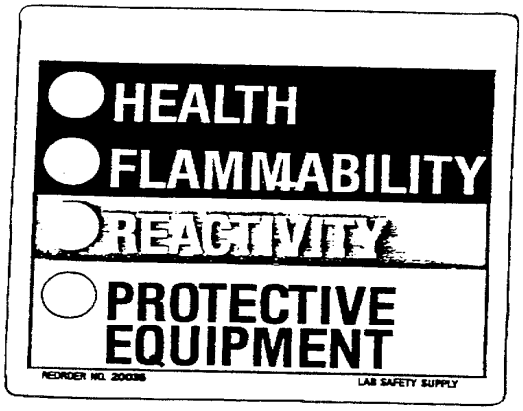


3. Each container of hazardous waste must have the proper hazard labels fixed. They should be placed on the top of the container and on two sides of the container opposite each other. Preferably the labels should be placed next to the labels described above. See page two of this attachment for complete shipping information for each waste stream.

Attachment 9 (continued)

Attachment 9 (continued)





APPENDIX E
DATA VALIDATION WORKSHEETS

VOLATILE ORGANICS

SOP # HW-24
Revision # 2
August 2008

USEPA
Hazardous Waste Support Branch
Validating Volatile Organic Compounds
By Gas Chromatography/Mass Spectrometry
SW-846 Method 8260B

Prepared by: George Karras Date: 8/22/08
George Karras, Chemist
Hazardous Waste Support Section

Prepared by: Russell Arnone Date: 8/25/08
Russell Arnone, Chemist
Hazardous Waste Support Section

Concurred by: Linda M. Mauel Date: 8/26/08
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 8/26/08
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: Russell Arnone Date: 08/11/09
Name

Reviewed by: _____ Date: _____
Name

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to the USEPA SW-846, Method 8260B December 1996. The validation methods and actions discussed in this document are based on the requirements set forth in USEPA SW-846, Method 8260B and Method 8000C, Rev 3, March 2003; and "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," January, 2005. This document covers technical as well as method specific problems; however situations may arise where data limitations must be assessed based on the reviewer's own professional judgement.

Summary

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 4.

The reviewer must prepare a detailed data assessment to be submitted along with the complete SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data, and contract non-compliance.

DEFINITIONS

Acronyms

BNA - base neutral acid(another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
CF - calibration factor
%D - percent difference
DCB -decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatography/electron capture detector
GC/MS - gas chromatography/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
ℓ - liter
mℓ - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCX -tetrachloro-m-xylene
TIC - tentatively identified compound

TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

NJ - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

LAB QUALIFIERS:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

YES NO N/A

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ LAB:_____

SITE NAME:_____

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format or CLP Forms Equivalent? [ ] ___ ___

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative, and/or cover letter signed release present? [ ] ___ ___

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [ ] ___ ___

ACTION: If not, note the effect on review of the data in the Data Assessment narrative.

II. VOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Reports, and/or Chain of Custodies from the field samplers present for all samples sign release present? [ ] ___ ___

ACTION: If no, contact the laboratory/sampling team for replacement of missing or illegible copies.

1.2 Is a sampling trip report present (if required)? [ ] ___ ___

1.3 Sample Conditions/Problems

YES NO N/A

1.3.1 Do the Traffic Reports, Chain of Custodies, or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ___ [ ] ___

ACTION: If all the VOA vials for a sample have air bubbles or the VOA vial analyzed had air bubbles, flag all positive results "J" and all non-detects "R".

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, flag all positive results "J" and all non-detects "R".

ACTION: If samples were not iced or if the ice was melted upon receipt at the laboratory and the temperature of the cooler was elevated (>10°C), flag all positive results "J" and all non-detects non"UJ".

2.0 Holding Times

2.1 Have any volatile holding times, determined from date of collection to date of analysis, been exceeded? ___ [ ] ___

The maximum holding time for aqueous samples is 14 days.

The maximum holding time for soils non aqueous samples is 14 days.

NOTE: If unpreserved, aqueous samples maintained at 4°C for aromatic hydrocarbons analysis must be analyzed within 7 days. If preserved with HCL acid to a pH<2 and stored at 4°C, then aqueous samples must be analyzed within 14 days from time of collection. For non-aqueous samples for volatile components that are frozen (less than 7°C) or are properly cooled (4°C ± 2°C) and perserved with $NaHSO_4$, the maximum holding time is 14 days from sample collection. If

YES NO N/A

uncertain about preservation, contact the laboratory /sampling team to determine whether or not samples were preserved.

ACTION: Qualify sample results according to Table 1:

Table 1. Holding Time Actions for Trace Volatile Analysis

Matrix	Preserved	Criteria	Action	
			Detected Associated Compounds	Non-Detected Associated Compounds
Aqueous	No	≤7 days	No qualifications	
	No	> 7 days	J	R
	Yes	≤14 days	No qualifications	
	Yes	> 14 days	J	R
Non Aqueous	No	≤ 14 days	J	R
	Yes	≤ 14 days	No qualifications	
	Yes/No	> 14 days	J	R

3.0 Surrogate Recovery (CLP Form II Equivalent)

3.1 Have the volatile surrogate recoveries been listed on Surrogate Recovery forms for each of the following matrices:

a. Water [ ] ___ ___

b. Soil [ ] ___ ___

3.2 If so, are all the samples listed on the appropriate Surrogate Recovery forms for each matrix:

a. Water [ ] ___ ___

b. Soil [ ] ___ ___

ACTION: If large errors exist, deliverables are unavailable or information is missing, document the effect(s) in Data

YES NO N/A

Assessments and contact the laboratory/project officer/appropriate official for an explanation /resubmittal, make any necessary corrections and document effect in the Data Assessment.

3.3 Were the surrogate recovery limits followed per Table 2. If Table 2 criteria were not followed, the laboratory may use in-house performance criteria (per SW-846, Method 8000C, section 9.7). Other compounds may be used as surrogates, depending upon the analysis requirements. [ ] ___ ___

Table 2. Surrogate Spike Recovery Limits for Water and Soil/Sediments

DMC	Recovery Limits (%)Water	Recovery Limits Soil/Sediment
4-Bromofluorobenzene	80-120	70-130
Dibromofluoromethane	80-120	70-130
Toluene-d_8	80-120	70-130
Dichloroethane-d_4	80-120	70-130

Note: Use above table if laboratory did not provide in house recovery criteria.

Note: Other compounds may be used as surrogated depending upon the analysis requirements.

3.4 Were outliers marked correctly with an asterisk? [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

3.5 Were one or more volatile surrogate recoveries out of specification for any sample or method blank. Table 2. [ ] ___ ___

If yes, were samples reanalyzed? [ ] ___ ___

Were method blanks reanalyzed? [ ] ___ ___

YES NO N/A

ACTION: If all surrogate recoveries are > 10% but 1 or more compounds do not meet method specifications:

1. Flag all positive results as estimated ("J").
2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.
3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects, but qualify positive results as estimated "J".

If any surrogate has a recovery of < 10%:

1. Positive results are qualified with ("J").
2. Non-detects for that should be qualified as unusable ("R").

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. The basic concern is whether the blank problems represent an isolated problem with the blank alone or whether there is a fundamental problem with the analytical process. If one or more samples in the batch show acceptable surrogate recoveries, the reviewer may choose the blank problem to be an isolated occurrence.

3.6 Are there any transcription/calculation errors between raw data and reported data? [ ] ___ ___

ACTION: If large errors exist, take action as specified in section 3.2 above.

4.0 <u>Laboratory Control Sample(Form III/Equivalent)</u>

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples of a similar matrix, per SDG. [ ] ___ ___

YES NO N/A

Note: LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume.

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation /resubmittals. Make note in the data assessment.

4.2 Were the Laboratory Control Samples analyzed at the required frequency for each of the following matrices:

A. Water [ ] ___ ___

B. Soil [ ] ___ ___

C. Med Soil [ ] ___ ___

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

4.3 Have in house LCS recovery limits been developed (Method 8000C, Sect 9.7). [ ] ___ ___

4.4 If in house limits are not developed, are LCS acceptance recovery limits between 70 - 130% (Method 8000c Sect 9.5)? [ ] ___ ___

4.5 Were one or more of the volatile LCS recoveries outside the in house laboratory recovery criteria for spiked analytes? If in house limits are not present use 70 - 130% recovery limits. [ ] ___ ___

YES NO N/A

Table 3. LCS Actions for Volatile Analysis

Criteria	Action	
	Detected Spiked Compounds	**Non-Detected Spiked Compounds**
%R > Upper Acceptance Limit	J	No Qualifiers
%R < Lower Acceptance Limit	J	UJ
Lower Acceptance Limit ≤ %R	No Qualifications	

5.0 Matrix Spikes(Form III or equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix? [ ] ___ ___

NOTE: The laboratory should use one matrix spike and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If the sample is not expected to contain target analytes, a MS/MSD should be analyzed (SW-846, Method 8260B, Sect 8.4.2).

5.2 Have MS/MD or MS/MSD results been summarized on modified CLP Form III? [ ] ___ ___

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples

YES NO N/A

of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month [page 8000C, section 9.5.])

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

Note: The LCS is spiked with the same analytes at the same concentrations as the matrix spike (SW-846 8000C, Section 9.5). If different make note in data assessment. Matrix/LCS spiking standards should be prepared from volatile organic compounds which are representative of the compounds being investigating. At a minimum, the matrix spike should include 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. The concentration of the LCS should be determined as described SW-Method 8000C Section 9.5.

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 Have in house MS recovery limits been developed (Method 8000C, Sect 9.7)for each matrix. [ ] ___ ___

5.5 Were one or more of the volatile MS/MSD recoveries outside of the in-house laboratory recovery criteria for spiked analytes? If none are present, then use 70-130% recovery as per SW-846, 8000C, Sect. 9.5.4. [ ] ___ ___

ACTION: Circle all outliers with a red pencil.

NOTE: If any individual % recovery in the MS (or MSD) falls outside the designated range for recovery the reviewer should determine if there is a matrix effect. A matrix effect is indicated if the LCS data are within limits but the MS data exceeds the limits.

YES NO N/A

NOTE: No qualification of data is necessary on MS and MSD data alone. However, using informed professional judgement, the data reviewer may use MS and MSD results in conjunction with other QC criteria to determine the need for some qualification.

Note: The data reviewer should first try to determine to what extent the results of the MS and MSD affect the associated data. This determination should be made with regard to he MS and MSD sample itself, as well as specific analytes for all samples associated with the MS and MSD.

Note: In those instances where it can be determine that the results of the MS and MSD affect only the sample spiked, limit qualification to this sample only. However, it may be determined through the MS and MSD results that a laboratory is having a systematic problem in the analysis of one or more analytes that affect all associated samples, and the reviewer must use professional judgement to qualify the data from all associated samples.

Note: The reviewer must use professional judgement to determine the need for qualification of non-spiked compounds.

ACTION: Follow criteria in Table 4 when professional judgement deems qualification of sample.

Table 4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) Actions for Volatile Analysis

Criteria	Action	
	Detected Spiked Compounds	Non-Detected Spiked Compounds
%R > Upper Acceptance Limit	J	No Qualifiers
%R < Lower Acceptance Limit	J	UJ
Lower Acceptance Limit ≤ %R	No Qualifications	

YES NO N/A

6.0 Blank (CLP Form IV Equivalent)

6.1 Is the Method Blank Summary form present? [ ] ___ ___

6.2 Frequency of Analysis: Has a method blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch? [ ] ___ ___

6.3 Has a method blank been analyzed for each GC/MS system used ? [ ] ___ ___

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject ® all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.4 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for volatile organic compounds? [ ] ___ ___

7.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.

[ ] ___ ___

	YES	NO	N/A
7.2 Do any field/rinse blanks have positive volatile organic compound results?	___	[ ]	___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 5 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

Table 5. Volatile Organic Analysis Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Storage, Field, Trip, Instrument**	Detects	Not detected	No qualification
	< CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgement
	> CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U, or qualify the data as unusable R
		≥ CRQL and ≥ blank contamination	Use professional judgement
	= CRQL*	< CRQL	Report CRQL value with a U
		≥ CRQL	Use professional judgement
	Gross contam-ination	Detects	Qualify results as unusable R

* 2x the CRQL for methylene chloride, 2-butanone, and acetone

** Qualifications based on instrument blank results affect only the sample analyzed immediately after the sample that has target compounds that exceed the calibration range or non-target compounds that exceed 100 ug/L.

NOTE: If gross blank contamination exists(e.g., saturated peaks, "hump-o-grams," "junk" peaks), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference. Non-detected volatile organic target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

YES NO N/A

7.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 GC/MS Apparatus and Materials

8.1 Did the lab use the proper gas chromatographic column(s) for analysis of volatiles by Method 8260B? Check raw data, instrument logs or contact the lab to determine what type of column(s) was (were) used. [ ] ___ ___

NOTE: For the analysis of volatiles, the method requires the use of 60 m. x 0.75 mm capillary column, coated with VOCOL(Supelco) or equivalent column. (see SW-846, page 8260B-7, section 4.9.2)

ACTION: If the specified column, or equivalent, was not used, document the effects in the Data Assessment. Use professional judgement to determine the acceptability of the data.

9.0 GC/MS Instrument Performance Check (CLP Form V Equivalent)

9.1 Are the GC/MS Instrument Performance Check forms present for Bromofluorobenzene (BFB), and do these forms list the associated samples with date/time analyzed? [ ] ___ ___

9.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the BFB provided for each twelve hour shift? [ ] ___ ___

9.3 Has an instrument performance check solution (BFB)

YES NO N/A

been analyzed for every twelve hours of sample analysis per instrument?(see Table 4, SW-846, page 8260B-36) [ ] ___ ___

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS GC/MS tuning data are available.

ACTION: If the laboratory/project officer cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable, "R".

9.4 Have the ion abundances been normalized to m/z 95? [ ] ___ ___

9.5 Have the ion abundance criteria been met for each instrument used? [ ] ___ ___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

ACTION: If ion abundance criteria are not met, take action as specified in section 3.2.

9.6 Are there any transcription/calculation errors between mass lists and reported values? (Check at least two values but if errors are found, check more.) ___ [ ] ___

9.7 Have the appropriate number of significant figures (two) been reported? [ ] ___ ___

ACTION: If large errors exist, take action as specified in section 3.2.

9.8 Are the spectra of the mass calibration compounds acceptable. [ ] ___ ___

ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.

YES NO N/A

10.0 Target Analytes (CLP Form I Equivalent)

10.1 Are the Organic Analysis reporting forms present with required header information on each page, for each of the following:

a. Samples and/or fractions as appropriate [ ] ___ ___

b. Matrix spikes and matrix spike duplicates [ ] ___ ___

c. Blanks [ ] ___ ___

d. Laboratory Control Samples [ ] ___ ___

10.2 Are the reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?

a. Samples and/or fractions as appropriate [ ] ___ ___

b. Matrix spikes and matrix spike duplicates (Mass spectra not required) [ ] ___ ___

c. Blanks [ ] ___ ___

d. Laboratory Control Samples [ ] ___ ___

ACTION: If any data are missing, take action specified in 3.2 above.

10.3 Is chromatographic performance acceptable with respect to:

Baseline stability? [ ] ___ ___

	YES	NO	N/A
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___

Other:_____

ACTION: Use professional judgement to determine the acceptability of the data.

10.4 Are the lab-generated standard mass spectra of identified volatile compounds present for each sample? [ ] ___ ___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the Data Assessment. If spectra are missing, contact the lab for missing spectra.

10.5 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration? [ ] ___ ___

10.6 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? [ ] ___ ___

10.7 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum? [ ] ___ ___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected ("R"), flagged ("N") - Presumptive evidence of the presence of the compound) or changed to non detected ("U") at the calculated detection limit. In order to be

YES NO N/A

positively identified, the data must comply with the criteria listed in 9.6, 9.7, and 9.8.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

11.0 Tentatively Identified Compounds (TIC) (CLP Form I/TIC Equivalent)

11.1 If Tentatively Identified Compound were required for this project, are all Tentatively Identified Compound reporting forms present; and do listed TICs include scan number or retention time, estimated concentration and a qualifier? [ ] ___ ___

NOTE: Add "N" qualifier to all TICs which have CAS number, if missing.

NOTE: Have the project officer/appropriate official check the project plan to determine if lab was required to identify non-target analytes (SW-846, page 8260B-23, Sect. 7.6.2).

11.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:

a. Samples and/or fractions as appropriate [ ] ___ ___

b. Blanks [ ] ___ ___

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by a CAS#.

NOTE: If TICs are present in the associated blanks take action as specified in section 3.2 above.

YES NO N/A

11.3 Are any priority pollutants listed as TIC compounds (i.e., an BNA compound listed as a VOA TIC)? [ ] ___ ___

ACTION: 1. Flag with "R" any target compound listed as a TIC.

2. Make sure all rejected compounds are properly reported if they are target compounds.

11.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum? [ ] ___ ___

11.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%? [ ] ___ ___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate. Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R". (Common lab contaminants: CO_2(M/E 44), Siloxanes (M/E 73), Hexane, Aldol Condensation Products, Solvent Preservatives, and related byproducts).

12.0 Compound Quantitation and Reported Detection Limits

12.1 Are there any transcription/calculation errors in organic analysis reporting form results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and average initial RRF/CF were used to calculate organic analysis reporting form result. Were any errors found? [ ] ___ ___

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be

YES NO N/A

reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

12.2 Are the method CRQL's adjusted to reflect sample dilutions and, for soils, sample moisture? [ ] ___ ___

ACTION: If errors are large, take action as specified in section 3.2 above.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC accedence dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original reporting form (if present) and substituting the data from the analysis of the diluted sample. Specify which organic analysis reporting form is to be used, then draw a red "X" across the entire page of all reporting forms that should not be used, including any in the summary package.

13.0 Standards Data (GC/MS)

13.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant Reports) present for initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in section 3.2 above.

14.0 GC/MS Initial Calibration (CLP Form VI Equivalent)

YES NO N/A

14.1 Are the Initial Calibration reporting forms present and complete for the volatile fraction? [ ] ___ ___

ACTION: If any calibration forms or standard raw data are missing, take action specified in section 3.2 above.

ACTION: If the percent relative standard deviation (% RSD) is > 20%, (8000C-39)qualify positive results for that analyte "J". When % RSD > 90%,. Qualify all positive results for that analyte "J" and all non-detects results for that analyte "R".

14.2 Are all average RRFs > 0.050? [ ] ___ ___

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list. If individual RRF values reported are below the listed values document in the Data Assessment.

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

ACTION: Circle all outliers with red pencil.

ACTION: For any target analyte with average RRF < 0.05, or for the requirements for the 5 compounds in 14.2 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

14.3 Are response factors stable over the concentration range of the calibration. [ ] ___ ___

NOTE: (Method Requirement) For the following CCC compounds, the %RSD values must be ≤ 30.0%. If %RSD values reported are > 30.0% document in the Data Assessment.

YES NO N/A

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

ACTION: Circle all outliers with a red pencil.

ACTION: If the % RSD is > 20.0%, or > 30% for the 6 compounds in 14.3 above, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

14.4 Was the % RSD determined using RRF or CF? [ ] ___ ___

If no, what method was used to determine the linearity of the initial calibration? Document any effects to the case in the Data Assessment.

14.5 Are there any transcription/calculation errors in the reporting of RRF or % RSD? (Check at least two values but if errors are found, check more.) [ ] ___ ___

ACTION: Circle errors with a red pencil.

ACTION: If errors are large, take action as specified in section 3.2 above.

15.0 <u>GC/MS Calibration Verification (CLP Form VII Equivalent)</u>

YES NO N/A

15.1 Are the Calibration Verification reporting forms present and complete for all compounds of interest? [ ] ___ ___

15.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument? [ ] ___ ___

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

_____

ACTION: If any forms are missing or no calibration verification standard has been analyzed twelve hours prior to sample analysis, take action as specified in section 3.2 above. If calibration verification data are not available, flag all associated sample data as unusable ("R").

15.3 Was the % D determined from the calibration verification determined using RRF or CF? [ ] ___ ___

If no, what method was used to determine the calibration verification? Document any effects to the case in the Data Assessment.

15.4 Do any volatile compounds have a % D (difference or drift) between the initial and continuing RRF or CF which exceeds 20% (SW-846, page 8260B-19, section 7.4.5.2). [ ] ___ ___

NOTE: (Method Requirement) For the following CCC compounds, the %D values must be ≤ 20.0%. If %D values reported are > 20.0% document in the Data Assessment.

1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethylbenzene
Vinyl chloride

YES NO N/A

ACTION: Circle all outliers with a red pencil.

ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated, "J". When %D is above 90%, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

15.5 Do any volatile compounds have a RRF < 0.05? [ ] ___ ___

NOTE: (Method Requirement) For SPCC compounds, the individual RRF values must be ≥ the values in the following list for each calibration verification. If average RRF values reported are below the listed values document in the data assessment.

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

ACTION: Circle all outliers with a red pencil.

ACTION: If RRF < 0.05, or < the requirements for the 5 compounds is section 15.5 above, qualify all positive results for that analyte "J" and all non-detect results for that analyte "R".

NOTE: The above data qualification action applies regardless of method requirements.

16.0 Internal Standards (CLP Form VIII Equivalent)

16.1 Are the internal standard (IS) areas on the internal standard reporting forms of every sample and blank within the upper and lower limits (-50% to + 100%) for each initial mid-point calibration (SW-846, 8260B-20, Sect. 7.4.7)? [ ] ___ ___

YES NO N/A

ACTION: If errors are large or information is missing, take action as specified in section 3.2 above.

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area Lower Limit	Area Upper Limit
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

(Attach additional sheets if necessary.)

ACTION:

1. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results quantitated with this internal standard.

2. Do not qualify non-detects when the associated IS are counts area > + 100%.

3. If the IS area is below the lower limit (< -50%), qualify all associated non-detects (U-values) "J".

4. If extremely low area counts are reported (< -25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable "R" and positive results as estimated "J".

16.2 Are the retention times of all internal standards within 30 seconds of the associated initial mid-point calibration standard (SW-846, 8260B-20, Sect. 7.4.6)? [ ] ___ ___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

YES NO N/A

17.0 Field Duplicates

17.1 Were any field duplicates submitted for volatile analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the Data Assessment. However, if large differences exist, take action specified in section 3.2 above.

SEMIVOLATILE ORGANICS

US EPA
Hazardous Waste Support Branch
Validating Semivolatile Organic Compounds
By Gas Chromatography/Mass Spectrometry
SW-846 Method 8270D

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Annual Review

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YES NO N/A

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8270D" January 1998. Method 8270D is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in SW846 Method 8270D, Method 8000C and the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," January 2005. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 5.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications

Data reviewers must possess a working knowledge of SW846 Analytical Methods and National Functional Guidelines mentioned above.

YES NO N/A

DEFINITIONS

Acronyms

BNA - base neutral acid(another name for Semi Volatiles)
CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB -decachlorobiphenyl
DDD - dichlorodiphenyldichloroethane
DDE - dichlorodiphenylethane
DDT - dichlorodiphenyltrichloroethane
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
ℓ - liter
mℓ - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation Mixture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure

YES NO N/A

TCX -tetrachloro-m-xylene
TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

Data Qualifiers

U - The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J - The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N - The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN - The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ - The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R - The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

LAB QUALIFIERS:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

YES NO N/A

E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

I. PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ LAB:_____

SITE NAME:_____

1.0 Data Completeness and Deliverables

1.1 Has all data been submitted in CLP deliverable format? [ ] ___ ___

ACTION: If not, note the effect on review of the data in the data assessment narrative.

2.0 Cover Letter, SDG Narrative

2.1 Is a laboratory narrative or cover letter present? [ ] ___ ___

2.2 Are case number and SDG number(s) contained in the narrative or cover letter? [ ] ___ ___

YES NO N/A

II. SEMIVOLATILE ANALYSES

1.0 Traffic Reports and Laboratory Narrative

1.1 Are the Traffic Report Forms present for all samples? [ ] ___ ___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the Traffic Reports or Lab Narrative indicate any problems with sample receipt, condition of samples, analytical problems or special notations affecting the quality of the data? ___ [ ] ___

ACTION: If any sample analyzed as a soil, other than TCLP, contains 50%-90% water, all data should be flagged as estimated ("J"). If a soil sample, other than TCLP, contains more than 90% water, all non-detects data are qualified as unusable (R), and detects are flagged "J".

ACTION: If samples were not iced, or if the ice was melted upon arrival at the laboratory and the cooler temperature was elevated (10°C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times

2.1 Have any semivolatile technical holding times, determined from date of collection to date of extraction, been exceeded? ___ [ ] ___

Continuous extraction of water samples for semivolatile analysis must be started within **7** days of the date of collection. Soil/sediment samples must be extracted within **14** days of collection. Extracts must be analyzed within

YES NO N/A

40 days of the date of extraction.

Table of Holding Time Violations

Sample ID	Sample Matrix	Date Sampled	Date Lab Received	(See Traffic Report) Date Extracted	Date Analyzed

ACTION: If technical holding times are exceeded, flag all positive results as estimated ("J") and sample quantitation limits as estimated ("UJ"), and document in the narrative that holding times were exceeded.

If analyses were done more than 14 days beyond holding time, either on the first analysis or upon re analysis, the reviewer must use professional judgement to determine the reliability of the data and the effects of additional storage on the sample results. At a minimum, all results should be qualified "J", but the reviewer may determine that non-detect data are unusable ("R"). If holding times are exceeded by more than 28 days, all non-detect data are unusable (R).

	YES	NO	N/A
3.0 Surrogate Recovery (Form II/Equivalent)			
3.1 Have the semi volatile surrogate recoveries been listed on CLP Surrogate Recovery forms (Form II) for each of the following matrices:			
a. Low Water	[ ]	___	___
b. Low/Med Soil	[ ]	___	___
3.2 If so, are all the samples listed on the appropriate Surrogate Recovery Summary forms for each matrix:			
a. Low Water	[ ]	___	___
b. Low/Med Soil	[ ]	___	___

ACTION: If CLP deliverables are unavailable, document the effect(s) in data assessments. In some cases the lab may have to be contacted to obtain the data necessary to complete the validation.

3.3 Were outliers marked correctly with an asterisk?	[ ]	___	___

ACTION: Circle all outliers in red.

3.4 Were two or more base neutral OR acid surrogate recoveries out of specification for any sample or method blank (Reviewer should use lab in house recovery limits. Use surrogate recovery limits from USEPA National Functional Guidlines January 2005 page 130, if in house limits are not available. See Method 8000B-43 or 80000C-24).	[ ]	___	___

Note: Examine lab in house limits for reasonableness.

If yes, were samples re-analyzed?	[ ]	___	___

	YES	NO	N/A
Were method blanks re-analyzed?	[ ]	___	___

ACTION: If all surrogate recoveries are > 10% but two within the base-neutral or acid fraction do not meet method specifications, <u>for the affected fraction only (i.e. either base-neutral or acid compounds):</u>

1. Flag all positive results as estimated ("J").

2. Flag all non-detects as estimated detection limits ("UJ") when recoveries are less than the lower acceptance limit.

3. If recoveries are greater than the upper acceptance limit, do not qualify non-detects.

If any base-neutral <u>or</u> acid surrogate has a recovery of < 10%:

1. Positive results for the fraction with < 10% surrogate recovery are qualified with "J".

2. Non-detects for that fraction should be qualified as unusable (R) .

NOTE: Professional judgement should be used to qualify data that have method blank surrogate recoveries out of specification in both original and reanalyses. Check the internal standard areas.

3.5 Are there any transcription/calculation errors between raw data and Form II?	___	[ ]	___

ACTION: If large errors exist, call lab for explanation/resubmittal, make any necessary corrections and document

YES NO N/A

effect in data assessments.

4.0 Matrix Spikes (Form III/Equivalent)

4.1 Have the semivolatile Matrix Spike and Matrix Spike Duplicate/or duplicate unspiked Sample recoveries been listed on the Recovery Form (Form III)? [ ] ___ ___

NOTE: Method 3500B/page 4 states the spiking compounds:

Base/neutrals	Acids
1,2,4-Trichlorobenzene	Pentachlorophenol
Acenaphthene	Phenol
2,4-Dinitrotoluene	2-Chlorophenol
Pyrene	4-Chloro-3-methylphenol
N-Nitroso-di-n-propylamine	4-Nitrophenol
1,4-Dichlorobenzene	

Note: Some projects may require the spiking of specific compounds of interest.

Note: See Method 8270D-sec 8.4.2 for deciding on whether to prepare and analyze duplicate samples or a martix spike/matrix spike duplicate. If samples are expected to contain target analytes, then laboratory may use one matrix spike and a duplicate analysis of an unspiked field sample. If samples are not expected to contain target analytes, laboratory should use a matrix spike and matrix spike duplicate pair.

4.2 Were matrix spikes analyzed at the required frequency for each of the following matrices:

a. Low Water [ ] ___ ___

b. Low Solid [ ] ___ ___

c. Med Solid [ ] ___ ___

YES NO N/A

ACTION: If any matrix spike data are missing, take the action specified in 3.2 above. It may be necessary to contact the lab to obtain the required data.

NOTE: If the data has not been reported on CLP equivalent form, then the laboratory must provide the information necessary to evaluate the spike recoveries in the MS and MSD. The required data which should have been provided by the lab include the analytes and concentrations used for spiking, background concentrations of the spiked analytes (i.e., concentrations in unspiked sample), methods and equations used to calculate the QC acceptance criteria for the spiked analytes, percent recovery data for all spiked analytes.

The data reviewer must verify that all reported equations and percent recoveries are correct before proceeding to the next section.

4.3 Were matrix spikes performed at concentration equal to 100ug/L for acid compounds, and 200ug/l for base compounds (Method 3500B-4), or those specified in project plan. [ ] ___ ___

4.4 How many semivolatile spike recoveries are outside Laboratory in house MS/MSD recovery limits (use recovery limits values in Method 8270D-43&44 Table 6 if in house values not available).

Water

____ out of ____

Solids

____ out of ____

YES NO N/A

4.5 How many RPD's for matrix spike and matrix spike duplicate recoveries are outside QC limits?

Water Solids

____ out of ____ ____ out of ____

ACTION: Circle all outliers with red pencil.

ACTION: No action is taken on MS/MSD data alone. However, using informed professional judgement, the data reviewer may use the matrix spike and matrix spike duplicate results in conjunction with other QC criteria to determine the need for some qualification of the data.

4.6 Was a Laboratory Control Sample (LCS) analyzed with each analytical batch? [ ] ___ ___

NOTE: When the results of the matrix spike analysis indicate a potential problem due to the sample matrix itself, the LCS results are used to verify that the laboratory can perform the analysis in a clean matrix.

5.0 Blanks (Form IV/Equivalent)

5.1 Is the Method Blank Summary (Form IV) present? [ ] ___ ___

5.2 Frequency of Analysis:

Has a reagent/method blank analysis been reported per 20 samples of similar matrix, or concentration level, and for each extraction batch? [ ] ___ ___

5.3 Has a method blank been analyzed either after

	YES	NO	N/A
the calibration standard or at any other time during the analytical shift for each GC/MS system used ?	[ ]	___	___

ACTION: If any method blank data are missing, call lab for explanation/resubmittal. If not available, use professional judgement to determine if the associated sample data should be qualified.

5.4 Chromatography: review the blank raw data - chromatograms (RICs), quant reports or data system printouts and spectra.			
Is the chromatographic performance (baseline stability) for each instrument acceptable for the semivolatiles?	[ ]	___	___

ACTION: Use professional judgement to determine the effect on the data.

6.0 Contamination

NOTE: "Water blanks", "drill blanks" and "distilled water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

6.1 Do any method/instrument/reagent blanks have positive results for target analytes and/or TICs? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample dilution factor and corrected for percent moisture where necessary.	___	[ ]	___
6.2 Do any field/rinse/ blanks have positive results for target analytes and/or TICs (if required, see section 10 below)?	___	[ ]	___

YES NO N/A

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case) must be used to qualify data. Blanks may not be qualified because of contamination in another blank. Field Blanks must be qualified for outlying surrogates, poor spectra, instrument performance or calibration QC problems.

ACTION: Follow the directions in the table below to qualify sample results due to contamination. Use the largest value from all the associated blanks. If gross contamination exists, all data in the associated samples should be qualified as unusable (R).

YES NO N/A

Blank Action for Semivolatile Analyses

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Field	Detects	Not detected	No qualification required
	< CRQL *	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	= CRQL *	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification required
	> CRQL *	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report concentration of sample with a U
		≥ CRQL and ≥ blank contamination	No qualification required

NOTE: Analytes qualified "U" for blank contamination are still considered as "hits" when qualifying for calibration criteria.

NOTE: If the laboratory did not report TIC analyses, check the project plans to verify whether or not it was required.

6.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

6.4 Was a instrument blank analyzed after each sample/dilution which contained a target compound

	YES	NO	N/A
that exceeded the initial calibration range.	[ ]	___	___
6.5 Does the instrument blank have positive results for target analytes and/or TICs?	___	[ ]	___

Note: Use professional judgement to determine if carryover occurred and qualify analytes accordingly.

7.0 GC/MS Apparatus and Materials

	YES	NO	N/A
7.1 Did the lab use the proper gas chromatographic column for analysis of semivolatiles by Method 8270D? Check raw data, instrument logs or contact the lab to determine what type of column was used. The method requires the use of 30 m x 0.25 mm ID (or 0.32 mm ID), silicone-coated, fused silica, capillary column.	[ ]	___	___

ACTION: If the specified column, or equivalent, was not used, document the effects in the data assessment. Use professional judgement to determine the acceptability of the data.

8.0 GC/MS Instrument Performance Check (Form V/Equivalent)

	YES	NO	N/A
8.1 Are the GC/MS Instrument Performance Check Forms (Form V) present for decafluorotriphenylphosphine (DFTPP)?	[ ]	___	___

NOTE: The performance solution should also contain 4,4-DDT, pentachlorophenol, and benzidine to verify injection port inertness and column performance. The degradation of DDT to DDE and DDD must be less than 20% total and the response of pentachlorophenol and benzidine should be within normal ranges for these compounds (based upon lab experience) and show no peak degradation or tailing before samples are analyzed. (see section 5.5

	YES	NO	N/A
page 8270D-12).			
8.2 Are the enhanced bar graph spectrum and mass/charge (m/z) listing for the DFTPP provided for each twelve hour shift?	[ ]	___	___
8.3 Has an instrument performance check solution been analyzed for every twelve hours of sample analysis per instrument?	[ ]	___	___

ACTION: List date, time, instrument ID, and sample analyses for which no associated GC/MS tuning data are available.

DATE	TIME	INSTRUMENT	SAMPLE NUMBERS
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

ACTION: If lab cannot provide missing data, reject ("R") all data generated outside an acceptable twelve hour calibration interval.

ACTION: If mass assignment is in error, flag all associated sample data as unusable (R).

8.4 Have the ion abundances been normalized to m/z 198?	[ ]	___	___
8.5 Have the ion abundance criteria been met for each instrument used?	[ ]	___	___

ACTION: List all data which do not meet ion abundance criteria (attach a separate sheet).

	YES	NO	N/A
ACTION: If ion abundance criteria are not met, take action specified in section 3.2			
8.6 Are there any transcription/calculation errors between mass lists and Form Vs? (Check at least two values but if errors are found, check more.)	___	[ ]	___
8.7 Have the appropriate number of significant figures (two) been reported?	[ ]	___	___
ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document effect in data assessments.			
8.8 Are the spectra of the mass calibration compound acceptable?	[ ]	___	___
ACTION: Use professional judgement to determine whether associated data should be accepted, qualified, or rejected.			
9.0 Target Analytes			
9.1 Are the Organic Analysis Data Sheets (Form I) present with required header information on each page, for each of the following:			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates	[ ]	___	___
c. Blanks	[ ]	___	___
9.2 Has any special cleanup, such as GPC, been performed on all soil/sediment sample extracts (see section 7.2, page 8270D-14)?	[ ]	___	___

	YES	NO	N/A
ACTION: If data suggests that extract cleanup was not performed, use professional judgement. Make note in the data assessment narrative.			
9.3 Are the Reconstructed Ion Chromatograms, mass spectra for the identified compounds, and the data system printouts (Quant Reports) included in the sample package for each of the following?			
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Matrix spikes and matrix spike duplicates (Mass spectra not required)	[ ]	___	___
c. Blanks	[ ]	___	___
ACTION: If any data are missing, take action specified in 3.2 above.			
9.4 Are the response factors shown in the Quant Report?	[ ]	___	___
9.5 Is chromatographic performance acceptable with respect to:			
Baseline stability?	[ ]	___	___
Resolution?	[ ]	___	___
Peak shape?	[ ]	___	___
Full-scale graph (attenuation)?	[ ]	___	___
Other:_____	[ ]	___	___
ACTION: Use professional judgement to determine the acceptability of the data.			
9.6 Are the lab-generated standard mass spectra of identified semivolatile compounds present for			

	YES	NO	N/A
each sample?	[ ]	___	___

ACTION: If any mass spectra are missing, take action specified in 3.2 above. If the lab does not generate their own standard spectra, make a note in the data assessment narrative. If spectra are missing, reject all positive data.

9.7 Is the RRT of each reported compound within 0.06 RRT units of the standard RRT in the continuing calibration?	[ ]	___	___
9.8 Are all ions present in the standard mass spectrum at a relative intensity greater than 10% (of the most abundant ion) also present in the sample mass spectrum?	[ ]	___	___
9.9 Do the relative intensities of the characteristic ions in the sample agree within ± 30% of the corresponding relative intensities in the reference spectrum?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of data. If it is determined that incorrect identifications were made, all such data should be rejected (R), flagged "N" (Presumptive evidence of the presence of the compound) or changed to not detected (U) at the calculated detection limit. In order to be positively identified, the data must comply with the criteria listed in 9.7, 9.8, and 9.9.

ACTION: When sample carry-over is a possibility, professional judgement should be used to determine if instrument cross-contamination has affected any positive compound identification.

	YES	NO	N/A

10.0 Tentatively Identified Compounds (TIC)

10.1 If Tentatively Identified Compounds were required for this project, are all Form Is, Part B present; and do listed TICs include scan number or retention time, estimated concentration and "JN" qualifier?

NOTE: Review sampling reports to determine if the lab was required to identify non target analytes (refer to section 7.6.2,page 8270D-21).

10.2 Are the mass spectra for the tentatively identified compounds and associated "best match" spectra included in the sample package for each of the following:	[ ]	___	___
a. Samples and/or fractions as appropriate	[ ]	___	___
b. Blanks	[ ]	___	___

ACTION: If any TIC data are missing, take action specified in 3.2 above.

ACTION: Add "JN" qualifier only to analytes identified by CAS #.

10.3 Are any target compounds from one fraction listed as TIC compounds in another (e.g., an acid compound listed as a base neutral TIC)?	___	[ ]	___

ACTION: i. Flag with "R" any target compound listed as a TIC.

ii. Make sure all rejected compounds are properly reported in the other fraction.

10.4 Are all ions present in the reference mass spectrum with a relative intensity greater than 10% (of the most abundant ion) also present in the

	YES	NO	N/A
sample mass spectrum?	[ ]	___	___
10.5 Do TIC and "best match" standard relative ion intensities agree within ± 20%?	[ ]	___	___

ACTION: Use professional judgement to determine acceptability of TIC identifications. If it is determined that an incorrect identification was made, change the identification to "unknown" or to some less specific identification (example: "C3 substituted benzene") as appropriate and remove "JN". Also, when a compound is not found in any blank, but is a suspected artifact of a common laboratory contaminant, the result should be qualified as unusable, "R."

11.0 Compound Quantitation and Reported Detection Limits

	YES	NO	N/A
11.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Verify that the correct internal standard, quantitation ion, and RRF were used to calculate Form I result. Were any errors found?	___	[ ]	___

NOTE: Structural isomers with similar mass spectra, but insufficient GC resolution (i.e. percent valley between the two peaks > 25%) should be reported as isomeric pairs. The reviewer should check the raw data to ensure that all such isomers were included in the quantitation (i.e., add the areas of the two coeluting peaks to calculate the total concentration).

	YES	NO	N/A
11.2 Are the method detection limits adjusted to reflect sample dilutions and, for soils, sample moisture?	[ ]	___	___

YES NO N/A

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest detection limits are used (unless a QC exceedance dictates the use of the higher detection limit from the diluted sample data). Replace concentrations that exceed the calibration range in the original analysis by crossing out the "E" and it's associated value on the original Form I (if present) and substituting the data from the analysis of the diluted sample. Specify which Form I is to be used, then draw a red " X" across the entire page of all Form I's that should not be used, including any in the summary package.

12.0 Standards Data (GC/MS)

12.1 Are the Reconstructed Ion Chromatograms, and data system printouts (Quant, Reports) present for initial and continuing calibration? [ ] ___ ___

ACTION: If any calibration standard data are missing, take action specified in 3.2 above.

13.0 GC/MS Initial Calibration (Form VI/Equivalent)

13.1 Is the Initial Calibration Form (Form VI/ Equivalent) present and complete for the semivolatile fraction? [ ] ___ ___

ACTION: If any calibration forms or standard row data are missing, take action specified in 3.2 above.

13.2 Are all base neutral or acid RRFs > 0.050? [ ] ___ ___

YES NO N/A

Check the **average RRFs** of the four System Performance Check Compounds (SPCCs): N-nitroso-di-n-propylamine, hexachlorocyclopentadiene, 2,4-dinitrophenol, and 4-nitrophenol. These compounds must have **average RRFs** greater than or equal to 0.05 before running samples and should not show any peak tailing.

ACTION: Circle all outliers in red.

ACTION: For any target analyte with **average RRF <0.05**

1. "R" all non-detects;

2. "J" all positive results.

13.3 Are response factors for base neutral or acid target analytes stable over the concentration range of the calibration (% Relative standard deviation [%RSD] < 20.0%)? [ ] ___ ___

NOTE: The % RSD for each individual Calibration Check Compound (CCC, Method 8270D-40 see Table 4) must be less than 30% before analysis can begin. If grater 30%, the lab must clean and recalibrate the instrument.

CALIBRATION CHECK COMPOUNDS

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
Diphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol

	YES	NO	N/A
Benzo(a)pyrene			

ACTION: If the %RSD for any CCC >30% and no corrective action taken, then "J" qualify all positive hits and "UJ" qualify all non-detects.

ACTION: Circle all outliers in red.

ACTION: If the % RSD is ≥ 20.0%, qualify positive results for that analyte "J" and non-detects using professional judgement. When RSD > 90%, flag all non- detect results for that analyte "R," unusable. Alternatively, the lab should calculate first or second order regression fit of the calibration curve and select the fit which introduces the least amount of error.

NOTE: Analytes previously qualified "U" due to blank contamination are still considered as "hits" when qualifying for calibration criteria.

	YES	NO	N/A
13.4 Did the laboratory calculate the calibration curve by the least squares regression fit?	___	[ ]	___
13.5 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or % RSD? (Check at least two values but if errors are found, check more.)	___	[ ]	___

ACTION: Circle Errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and note errors in data assessments.

	YES	NO	N/A
13.5 Do the target compounds for this SDG include Pesticides?	[ ]	___	___

	YES	NO	N/A
13.6 If the pesticide compounds include DDT, was the percent breakdown of DDT to DDD and DDE greater than 20%?	___	[ ]	___

ACTION: If DDT percent breakdown exceeds 20%:

i. Qualify all positive results for DDT with "J". If DDT was not detected, but DDD and DDE results are positive, qualify the quantitation limit for DDT as unusable, "R".

ii. Qualify all positive results for DDD and DDE as presumptively present at an approximate concentration "JN".

14.0 GC/MS Calibration Verification (Form VII/Equivalent)

14.1 Are the Calibration Verification Forms (Form VII) present and complete for all compounds of interest?	[ ]	___	___
14.2 Has a calibration verification standard been analyzed for every twelve hours of sample analysis per instrument?	[ ]	___	___

ACTION: List below all sample analyses that were not within twelve hours of a calibration verification analysis for each instrument used.

_____

_____

_____

ACTION: If any forms are missing or no calibration verification standard has been analyzed within twelve hours of every sample analysis,

	YES	NO	N/A
call lab for explanation/resubmittal. If continuing calibration data are not available, flag all associated sample data as unusable ("R").			
14.3 Do any of the SPCCs have an RRF <0.05?	___	[ ]	___
If YES, make a note in data assessment if the lab did not take corrective action specified in section 7.4.4, page 8270D-18.	[ ]	___	___
14.4 Do any of the CCCs have a %D between the initial and continuing RRF which exceeds 20.0%?			
ACTION: If yes, make a note in data assessment.			
14.5 Do any semivolatile compounds have a % Difference (% D) between the initial and continuing RRF which exceeds 20.0%?	___	[ ]	___
ACTION: Circle all outliers in red.			
ACTION: Qualify both positive results and non-detects for the outlier compound(s) as estimated (J). When %D is above 90%, qualify all non-detects for that analyte as "R", unusable.			
14.6 Do any semivolatile compounds have a RRF < 0.05?	___	[ ]	___
ACTION: Circle all outliers in red.			
ACTION: If RRF < 0.05, qualify as unusable ("R") associated non-detects and "J" associated positive values.			
14.7 Are there any transcription/calculation errors in the reporting of average response factors (RRF) or percent difference (%D) between initial and continuing RRFs? (Check at least two values but if errors are found, check more).	___	[ ]	___

YES NO N/A

ACTION: Circle errors in red.

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect(s) in the data assessments.

15.0 Internal Standards (Form VIII)

15.1 Are the internal standard areas (Form VIII) of every sample and blank within the upper and lower limits (-50% to + 100%) for each continuing calibration? [ ] ___ ___

ACTION: List each outlying internal standard below.

Sample ID	IS #	Area	LowerLimit	Upper Limit

(Attach additional sheets if necessary.)

Note: Check Table 5, 8270D-41 for associated analytes.

ACTION: i. If the internal standard area count is outside the upper or lower limit, flag with "J" all positive results and non-detects (U values) quantitated with this internal standard.

ii. Non-detects associated with IS > 100% should not be qualified.

	YES	NO	N/A
iii. If the IS area is below the lower limit (<50%), qualify all associated non-detects (U-values) "J". If extremely low area counts are reported (<25%) or if performance exhibits a major abrupt drop off, flag all associated non-detects as unusable (R).			
15.2 Are the retention times of all internal standards within 30 seconds of the associated calibration standard?	[ ]	___	___

ACTION: Professional judgement should be used to qualify data if the retention times differ by more than 30 seconds.

16.0 Laboratory Control Samples (LCS)

16.1 Were any LCS samples run in order to verify analytes which failed criteria for spike recovery?	[ ]	___	___
16.2 Did the lab spike LCS sample spiked with the same analytes and the same concentrations as the matrix spike?	[ ]	___	___
16.3 Were the mean and standard deviation of all analytes within the QC acceptance ranges as shown in Table 6, 8270D-43?	[ ]	___	___

ACTION: If the recovery of any analyte falls out of the designated range, the analytical results for that compound is suspect and should be qualified "J" in the unspiked samples.

17.0 Field Duplicates

17.1 Were any field duplicates submitted for semivolatile analysis?	[ ]	___	___

YES NO N/A

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, identification of field duplicates should be confirmed by contacting the sampler.

DIESEL RANGE ORGANICS

This SDG contains DRO results SW-846 method 8015B. Region II validation guidelines were used as applicable, however, the Region has not developed an SOP for this method so these worksheets are used as an alternative.

Holding Times

Sampling Date:
Received Date:
Preparation Date:
Analysis Dates:

7-day water or 14 day soil sample holding time was applied based on SW-846 recommendation, cooler temps *were/were not* acceptable. Appropriate preservation *was/was not* used.

All sample extraction and analysis holding time requirements *were/were not* met for all samples in this SDG. Qualifications needed:

Calibrations

_____-point calibration curves were analyzed for both the target and the surrogate compound on the _____ instrument(s) used to analyze these samples. The CFs and %RSD's were calculated and *did/did not* meet criteria for both the target the surrogate compound. Continuing calibration standards *were/were not* analyzed per the method. All %Ds *were/were not* within QC limits in all CCVs. These samples were analyzed on ____instrument(s) and _____ sequence(s). Qualifications needed:

Blank Summary

Blank qualification guidelines:

- No action is taken if a compound is found in the blank but not in the sample.
- **Sample weight, volume or dilution factor must be taken into consideration when applying criteria.**
- Apply the same data validation guidelines to any associated method, trip, rinse and field blanks and all associated samples.
- Qualification/Action codes:

U -	The blank contamination concentration is ≤ RL or > RL and sample result is < RL. Result is qualified as U at the RL.
U -	The blank contamination concentration is >RL and sample result >RL but < blank contamination concentration. Result is qualified as U at reported concentration.
NA	The sample is greater than the RL when the blank contamination concentration is < RL or the sample result is greater than the blank contamination concentration when the blank contamination concentration is >RL.

Please note: the LOD = RL for this project.

Blank Contamination and Qualification Summaries

Blank ID	Compound	Concentration	LOD

Associated samples and required qualifications are noted in the following table.

Sample ID	Compound	Q Flag

Surrogate Recoveries Summary

All surrogate recoveries *were/were not* acceptable in these samples. Qualifications needed:

Matrix Spike/Matrix Spike Duplicate Summary

The MS/MSD pair submitted in this SDG exhibited acceptable/unacceptable recoveries and RPDs. LCS recoveries *were/were not* acceptable. Qualifications needed:

Field Duplicate Sample Summary

Sample ID: Duplicate Sample ID:

Compound	Sample Conc.	Duplicate Conc.	RPD
DRO			

Comments:

Sample Result Verification

Specific Comments:

Raw data was verified. *The samples required manual integration for the target compounds. No positive results were reported in the field samples.* Raw data and quantitation calculations were verified.

Reviewer_____ **Date:** _____

SAMPLE CALCULATION

Sample ID: Sample XYZ
Standard ID: ICAL, _____, VARIAN _____
Compound: DRO
Concentration: 381.3E mg/Kg

	Water (mg/L)	Soil (mg/Kg)
Area of Compound		23638548
CF of Compound		3453054
Final Volume		1000
Dilution Factor		1
GPC Factor	NA	1
Injection Volume		1
Weight of Sample	NA	20
Initial Volume of Sample		NA
% Solids Factor	NA	0.898
Concentration	#DIV/0!	381.163

Initial Calibration Date: _____ ICAL on Instrument _____

RRF and %RSD Calculations:

Compound Name: Diesel, Level 2
Lab Value: 3102746

Area of Compound	1551373
Conc. of Compound	0.5
Calculated CF	3102746

Compound Name: DRO
Lab Value: 15.579180

CF of STD 1	3753830.000000
CF of STD 2	3102746.000000
CF of STD 3	2689818.000000
CF of STD 4	3733762.000000
CF of STD 5	3985112.000000
CF of STD 6	
CF of STD 7	
Average CF	3453053.600000
Calculated % RSD	15.579187

Continuing Calibration File ID: Date/Time:_____, File ID _____

RRF and %D Calculations:

Compound Name: DRO
Lab Value: 3498423

Area of Compound	8746059
Conc. of Compound	2.5
Calculated CF	3498424

Compound Name: DRO
Lab Value: 1.3

Average CF	3453054
Calibration Check CF	3498423
Calculated % D	1.31

PESTICIDES

USEPA
Hazardous Waste Support Branch
Validating Pesticide Compounds
Organochlorine Pesticides By Gas Chromatography
SW-846 Method 8081B

Prepared by: George Karras Date: 12/8/06
George Karras, Chemist
Hazardous Waste Support Section

Prepared by: Russell Amone Date: 12-8-06
Russell Amone, Chemist
Hazardous Waste Support Section

Concurred by: Linda Mauel Date: 12/11/06
Linda Mauel, Chief
Hazardous Waste Support Section

Approved by: Robert Runyon Date: 12/11/06
Robert Runyon, Chief
Hazardous Waste Support Branch

Annual Review

Reviewed by: _____ Date: _____
Name

Reviewed by: _____ Date: _____
Name

INTRODUCTION

Scope and Applicability

This SOP offers detailed guidance in evaluating laboratory data generated according to "SW846-Method 8081B November 2000. Method 8081B is used to determine the concentration of pesticide compounds in extracts prepared from many types of solid waste matrices, soils, air sampling media and water samples. The validation methods and actions discussed in this document are based on the requirements set forth in SW846 Method 8081B, Method 8000C and the "USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," January 2005. This document covers technical problems specific to each fraction and sample matrix; however, situations may arise where data limitations must be assessed based on the reviewer's professional judgement.

Summary of Method

To ensure a thorough evaluation of each result in a data case, the reviewer must complete the checklist within this SOP, answering specific questions while performing the prescribed "ACTIONS" in each section. Qualifiers (or flags) are applied to questionable or unusable results as instructed. The data qualifiers discussed in this document are defined on page 4.

The reviewer must prepare a detailed data assessment to be submitted along with the completed SOP checklist. The Data Assessment must list all data qualifications, reasons for qualifications, instances of missing data and contract non-compliance.

Reviewer Qualifications

Data reviewers must possess a working knowledge of SW846 Analytical Methods and National Functional Guidelines mentioned above.

DEFINITIONS

Acronyms

CLP - Contract Laboratory Program
CRQL - Contract Required Quantitation Limit
%D - percent difference
DCB - decachlorobiphenyl
DoC - Date of Collection
GC - gas chromatography
GC/ECD - gas chromatograph/electron capture detector
GC/MS - gas chromatograph/mass spectrometer
GPC - gel permeation chromatography
IS - internal standard
kg - kilogram
µg - microgram
MS - matrix spike
MSD - matrix spike duplicate
ℓ - liter
mℓ - milliliter
PCB - Polychlorinated biphenyl
PE - performance evaluation
PEM - Performance Evaluation M_xture
QC - quality control
RAS - Routine Analytical Services
RIC - reconstructed ion chromatogram
RPD - relative percent difference
RRF - relative response factor
RRF - average relative response factor (from initial calibration)
RRT - relative retention time
RSD - relative standard deviation
RT - retention time
RSCC - Regional Sample Control Center
SDG - sample delivery group
SMC - system monitoring compound
SOP - standard operating procedure
SOW - Statement of Work
SVOA - semivolatile organic acid
TCL - Target Compound List
TCLP - Toxicity Characteristics Leachate Procedure
TCMX -tetrachloro-m-xylene
TIC - tentatively identified compound
TOPO - Task Order Project Officer
TPO - Technical Project Officer
VOA - Volatile organic
VTSR - Validated Time of Sample Receipt

Data Qualifiers

U- The analyte was analyzed for, but was not detected above the reported sample quantitation limit.

J- The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.

N- The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."

JN- The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.

UJ- The analyte was not detected above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.

R- The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte cannot be verified.

LAB QUALIFIERS:

D - The positive value is the result of an analysis at a secondary dilution factor.

B - The analyte is present in the associated method blank as well as in the sample. This qualifier has a different meaning when validating inorganic data.

E - The concentration of this analyte exceeds the calibration range of the instrument.

A - Indicates a Tentatively Identified Compound (TIC) is a suspected adol-condensation product.

X,Y,Z- Laboratory defined flags. The data reviewer must change these qualifiers during validation so that the data user may understand their impact on the data.

PACKAGE COMPLETENESS AND DELIVERABLES

CASE NUMBER:_____ SDG# _____
LAB:_____ SITE:_____

1.0 Data Completeness and Deliverables	YES	NO	N/A
1.1 Has all the data been submitted in CLP deliverable format?	[ ]	___	___
1.2 Have any missing deliverables been received and added to the data package?	[ ]	___	___

ACTION: Call lab for explanation/resubmittal of any missing deliverables. If lab cannot provide them, note the effect on review of the data in the reviewer narrative.

2.0 Cover Letter, SDG Narrative			
2.1 Is a laboratory narrative or cover letter present?	[ ]	___	___
2.2 Are the case number and/or SDG number contained in the narrative or cover letter?	[ ]	___	___

3.0 Data Validation Checklist			
3.1 Does this data package contain:			
Water data?	[ ]	___	___
Waste data?	[ ]	___	___
Soil/solid data?	[ ]	___	___

ORGANOCHLORINE PESTICIDE

	YES	NO	N/A
1.0 Traffic Reports and Laboratory Narrative			
1.1 Are traffic report and chain-of-custody forms present for all samples?	[ ]	___	___

ACTION: If no, contact lab for replacement of missing or illegible copies.

1.2 Do the traffic reports, chain-of-custody forms or SDG narrative indicate any problems with sample receipt, condition of the samples, analytical problems or special circumstances affecting the quality of the data?	___	[ ]	___

ACTION: If any sample analyzed as a soil, other than than TCLP, contains 50%-90% water, all data should be qualified as estimated, "J." If a soil sample, other than TCLP, contains more than 90% water, all non detects are qualified as unusable, "R", and positive results flagged "J".

ACTION: If samples were not iced or if the ice was melted upon arrival at the laboratory and the temperature of the cooler was elevated (> 10° C), flag all positive results "J" and all non-detects "UJ".

2.0 Holding Times			
2.1 Have any organochlorine pesticide technical holding times, determined from date of collection to date of extraction, been exceeded?	___	[ ]	___

Water and waste samples for organochlorine pesticide analysis must be extracted within 7 days of the date of collection. Extracts must be analyzed within 40 days of the date of extraction Soils and solid samples must be extracted within 14 days of collection and analyzed within 40 days of extraction.

ACTION: Qualify sample results according to Table 1.

Table 1. Holding Time Criteria

Matrix	Preserved	Criteria	Action	
			Detected compounds	Non-detected compounds
Aqueous	No	≤ 7 days(extraction) ≤ 40 days(analysis)	J*	UJ*
	No	> 7 days(extraction) > 40 days(analysis)	J*	UJ
	Yes	≤ 7 days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 7 days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R
Non-aqueous	No	≤ 14days(extraction) ≤ 40 days (analysis)	J*	UJ*
	No	> 14days(extraction) >40 days(analysis)	J	UJ
	Yes	≤ 14days(extraction) ≤ 40 days(analysis)	No qualification	
	Yes	> 14days(extraction) > 40 days(analysis)	J	UJ
	Yes/No	> 28 days (gross exceedance)	J	R

* only if cooler temperature exceeds 10°C; no action required if cooler temperature < 10°C.

YES NO N/A

3.0 Surrogate Recovery (Form II/Equivalent)

3.1 Were the recoveries of tetrachloro-m-xylene (TCMX) and decachlorobiphenyl (DCB) presented on CLP Surrogate Recovery Summary forms (Form II), or equivalent, for each of the following matrices?

a. Water/Waste [ ] ___ ___

b. Soil/Solid [ ] ___ ___

3.2 Are all the pesticide samples listed on the appropriate surrogate recovery form for each of the following matrices?

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

ACTION: Call lab for explanation/resubmittals. If missing deliverables are unavailable, document the effect in the data assessment.

3.3 Are all recovery limits for the surrogates TCMX and DCB between 30-150% for all samples, including MS and MSDs, LCSs and all blanks? [ ] ___ ___

Note: Reviewer shall use lab in-house recover limits if available. In-house criteria should be examined for reasonableness.

ACTION: Circle all outliers in red. Follow surrogate action Table 2.

3.5 Were surrogate retention times (RT) within the windows established during the initial 5-point analysis? [ ] ___ ___

ACTION: Follow surrogate action, Table 2 below.

YES NO N/A

Table 2. Surrogate Recovery Criteria

Criteria	Action	
	Detected Target Compounds	Non-detected Target Compounds
%R > 200%	J	Use professional judgement
150% < %R ≤ 200%	J	No qualification
30% ≤ %R ≤ 150%	No qualification	
10% ≤ %R < 30%	J	UJ
%R < 10% (sample dilution not a factor)	J	R
%R < 10% (sample dilution is a factor)	Use professional judgement	
RT out of RT window	Use professional judgement	
RT within RT window	No qualification	

3.6 Are there any transcription/calculation errors between raw data and Form II? [ ] ___ ___

ACTION: If large errors exist, call lab for explanation/resubmittal. Make any necessary corrections and document the effect in data assessments.

4.0 Laboratory Control Sample(LCS)

4.1 Is the LCS prepared, extracted, analyzed, and reported once for every 20 field samples. [ ] ___ ___

ACTION: If any Laboratory Control Sample data are missing, call the lab for explanation /resubmittals. Make note in the data assessment.

YES NO N/A

4.2 Were Laboratory Control Samples analyzed at the required concentration for all analytes of interest as specified in Table 3 below. [ ] ___ ___

Note: Use lab in-house criteria, if available.

Table 3. LCS Spiking Criteria

LCS Spike Compound	Spiking solution ug/l	Amount spiked to 100ml aqueous sample or 30g soil sample ml	Recovery Limits (%)
gamma-BHC	0.05	1	50-120
Heptachor epoxide	0.05	1	50-120
Dieldrin	0.01	1	30-130
4,4'-DDE	0.01	1	50-150
Endrin	0.01	1	50-120
Endosulfan sulfate	0.01	1	50-120
gamma-Chloradane	0.05	1	30-130
Tetrachloro-m-xylene(surrogate)	0.20	3	30-150
Decachlorobiphenyl (surrogate)	0.40	3	30-150

Note: The LCS might be spiked with the same analytes at the same concentration as the matrix spike.

ACTION: If Laboratory Control Samples were not analyzed at the required concentration or the required frequency, make note in the data assessment and use professional judgement to determined the affect on the data.

4.3 Do average recovery for each analyte meet the corresponding QC acceptance criteria listed in table above? [ ] ___ ___

YES NO N/A

ACTION: For LCS % recovery not meeting the required recovery, follow the required action in Table 4 below.

Table 4. LCS Recovery Criteria

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Compounds**
%R > Upper Acceptance Limit	J	No qualification
%R < Upper Acceptance Limit	J	R
Lower Acceptance Limit ≤ %R ≤ Upper Acceptance Limit	No qualifications	

5.0 Matrix Spikes (Form III/Equivalent)

5.1 Are all data for matrix spike and matrix duplicate or matrix spike duplicate (MS/MD or MS/MSD) present and complete for each matrix? [ ] ___ ___

NOTE: For soil and waste samples showing detectable amounts of organics, the lab may substitute replicate samples in place of the matrix spike (see page 8000B-40, section 8.5.3).

5.2 Have MS/MD or MS/MSD results been summarized on Form III/Equivalent? [ ] ___ ___

ACTION: If any data are missing take action as specified in section 3.2 above.

5.3 Were matrix spikes analyzed at the required frequency for each of the following matrices? (One MS/MD, MS/MSD or laboratory replicate must be performed for every 20 samples of similar matrix or concentration level. Laboratories analyzing one to ten samples per month are required to analyze at least one MS per month [page 8000B-39, section 8.5.])

YES NO N/A

a. Water [ ] ___ ___

b. Waste [ ] ___ ___

c. Soil/Solid [ ] ___ ___

ACTION: If any MS/MD, MS/MSD or replicate data are missing, take the action specified in 3.2 above.

5.4 We Were Matrix Spike Samples analyzed at the required concentration for all analytes of interest as specified in Table 5 below. [ ] ___ ___

Note: Spiking analytes may differ from those in Table 5. Check QA project plan or task order.

Table 5. Matrix Spiking Criteria

Matrix Spike Compound	Spiking solution ug/l	Amount spiked to 100ml aqueous sample or 30g soil sample ml
gamma-BHC	0.05	1
Heptachor	0.05	1
Aldrin	0.05	1
Dieldrin	1.0	1
Endrin	1.0	1
4,4'-DDT	1.0	1

Note: For aqueous organic extractable, the spike concentration should be:

1) For regulatory compliance monitoring - the regulatory concentration limit or 1 to 5 times the expected background concentration, whichever is higher;

2) For all other aqueous samples - the larger of either 1 to 5 x times the expected background

YES NO N/A

concentration, or the same as the QC check sample concentration (see section 4 above);

3) For soil/solid and waste samples - the recommended concentration is 20 times the estimated quantitation limit (EQL).

No action is taken based on MS or replicate data alone. However, using informed professional judgement, the data reviewer may use the matrix spike or laboratory replicate results in conjunction with other QC criteria and determine the need for some qualification of the data. In some instances it may be determined that only the replicate or spiked samples are affected. Alternatively, the data may suggest that the laboratory is having a systematic problem with one or more analytes, thereby affecting all associated samples.

5.5 Do average recovery for each analyte meet the corresponding QC acceptance criteria listed in Table 6 below. [ ] ___ ___

Note: Use lab in-house criteria, if available.

Table 6. Matrix Spike Recovery Criteria

Compound	% Recovery Water	RPD Water	% Recovery Soil	RPD Soil
gamma-BHC	56-123	0-15	46-127	0-50
Heptachor	40-13	0-20	35-130	0-31
Aldrin	40-120	0-22	34-132	0-43
Dieldrin	52-126	0-18	31-134	0-38
Endrin	56-121	0-21	42-139	0-45
4,4'-DDT	38-127	0-27	23-134	0-50

NOTE: The actual number of MS analytes depends on the number analytes being measured (e.g., total number of MS plus MSD compounds). If only chlordane or toxaphene are the analytes of

YES NO N/A

interest, the spiked sample should contain the most representative multi-component analyte.

ACTION: Follow the matrix spike actions (Table 7) for pesticide analyses.

Table 7. Matrix Spike Qualifying Criteria

Criteria	Action	
	Detected Associated Compounds	**Non-Detected Compounds**
%R or RPD > Upper Acceptance Limit	J	No qualification
20% R ≤ %R < Lower Acceptance Limit	J	UJ
%R < 20%	J	Use professional judgement
Lower Acceptance Limit ≤ %R; RPD ≤ Upper Acceptance Limit	No qualifications	

Note: When the results of the matrix spike analyses indicates a potential problem due to the sample matrix itself, the LCS results are used to verify the laboratory can perform analyses in a clean matrix.

6.0 Blanks (Form IV/Equivalent)

6.1 Was reagent blank data reported on Method Blank Summary form(s) (Form IV)?

6.2 Frequency of Analysis: Has a reagent blank been analyzed for every 20 (or less) samples of similar matrix or concentration or each extraction batch? [ ] ___ ___

Note: Method blank should be analyzed, either after the calibration standard or at any other time during the analytical shift.

YES NO N/A

ACTION: If any blank data are missing, take action as specified above (section 3.2). If blank data is not available, reject (R) all associated positive data. However, using professional judgement, the data reviewer may substitute field blank data for missing method blank data.

6.3 Chromatography: review the blank raw data - chromatograms, quant reports or data system printouts.

Is the chromatographic performance (baseline stability) for each instrument acceptable for pesticides? [ ] ___ ___

ACTION: Use professional judgement to determine the effect on the data.

7.0 Contamination

NOTE: "Water blanks", "distilled water blanks" and "drilling water blanks" are validated like any other sample and are not used to qualify the data. Do not confuse them with the other QC blanks discussed below.

7.1 Do any method/instrument/reagent/cleanup blanks have positive results for organochlorine pesticides? When applied as described below, the contaminant concentration in these blanks are multiplied by the sample Dilution Factor and corrected for % moisture when necessary. ___ [ ] ___

7.2 Do any field/rinse blanks have positive organochlorine pesticide results? ___ [ ] ___

ACTION: Prepare a list of the samples associated with each of the contaminated blanks. (Attach a separate sheet.)

NOTE: All field blank results associated to a particular group of samples (may exceed one per case or one per day) may be used to qualify data. Blanks may not be qualified because of contamination in

YES NO N/A

another blank. Field blanks must be qualified for surrogate, or calibration QC problems.

ACTION: Follow the directions in Table 8 below to qualify sample results due to contamination. Use the largest value from all the associated blanks.

Table 8. Blank Contamination Criteria

Blank Type	Blank Result	Sample Result	Action for Samples
Method, Clean up, Instrument, Field	Detects	Not detected	No qualification
	< CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	> CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL and < blank contamination	Report the concentration for the sample with a U
		≥ CRQL and ≥ blank contamination	No qualification
	= CRQL	< CRQL	Report CRQL value with a U
		≥ CRQL	No qualification
	Gross contamination	Detects	Qualify results as unusable R

Note: Analytes qualified "U" for blank contamination are treated as "hits" when qualifying the calibration criteria.

Note: When applied as described in Table 8 above, the contaminant concentration in the blank is multiplied by the sample dilution factor.

NOTE: If gross blank contamination exists(e.g., saturated peaks, "hump-o-grams", "junk peaks"), all affected positive compounds in the associated samples should be qualified as unusable "R", due to interference.

YES NO N/A

Non-detected pesticide target compounds do not require qualification unless the contamination is so high that it interferes with the analyses of non-detected compounds.

7.3 Are there field/rinse/equipment blanks associated with every sample? [ ] ___ ___

ACTION: For low level samples, note in data assessment that there is no associated field/rinse/equipment blank. Exception: samples taken from a drinking water tap do not have associated field blanks.

8.0 Gas Chromatography with Electron Capture Detector (GC/ECD)Instrument Performance Check (CLP Form VI and Form VII Equivalent)

8.1 Was the proper gas chromatographic column used for the analysis of organochlorine pesticides? Check raw data, instrument logs, or contact the lab to determine what type of columns were used. (See Method 8081B-8, section 4.2) [ ] ___ ___

8.2 If capillary columns were used, were they both wide bore (.53 mm ID) fused silica GC columns, such as DB-608 and DB-1701 or equivalent. Indicate the specific type of column used for:

column 1: _____

column 2: _____

ACTION: Note any changes to the suggested materials in section 8.1 above in the data assessment. Also note the impact (positive or negative) such changes have on the analytical results.

9.0 Calibration and GC Performance

9.1 Are the following Gas Chromatograms and Data Systems Printouts for both columns present for all samples, blanks, MS, replicates?

a. DDT/endrin breakdown check [ ] ___ ___

	YES	NO	N/A
b. toxaphene	[ ]	___	___
c. technical chlordane	[ ]	___	___
d. 5 pt. initial calibration standards	[ ]	___	___
e. calibration verification standards	[ ]	___	___
f. LCS	[ ]	___	___
g. Method blanks	[ ]	___	___

ACTION: If no, take action specified in 3.2 above.

9.2 Has a DDT/endrin breakdown check standard (at the mid-concentration level) been analyzed at the beginning of each analytical sequence on both columns (page 8081B-24, section 8.2.3)?	[ ]	___	___

ACTION: If no, take action as specified in 3.2 above.

9.3 Has the individual % breakdown exceeded 20.0% on either column for:

- 4,4' - DDT?	___	[ ]	___
- endrin?	___	[ ]	___

ACTION: If any % breakdown has failed the QC criteria in the breakdown check standard, qualify all sample analyses in the entire analytical sequence as described below.

a. If 4,4'-DDT breakdown is greater than 20.%:

i. Qualify all positive results for DDT with 'J". If DDT was not detected, but DDD and DDE are positive, then qualify the quantitation limit for DDT as unusable ("R").

ii. Qualify positive results for DDD and DDE as presumptively present at an approximated quantity ("NJ").

YES NO N/A

b. If endrin breakdown is greater than 20.0%:

i. Qualify all positive results for endrin with "J". If endrin was not detected, but endrin aldehyde and endrin ketone are positive, then qualify the quantitation limit for endrin as unusable ("R").

ii. Qualify positive results for endrin ketone and endrin aldehyde as presumptively present at an approximated quantity ("NJ").

9.4 Are data summary forms (containing calibration factors or response factors) for the initial 5 pt. calibration and daily calibration verification standards present and complete for each column and each analytical sequence? [ ] ___ ___

NOTE: If internal standard calibration procedure is used (page 8000B-16, section 7.4.2.2), then response factors must be used for %RSD calculations and compound quantitation. If, external standard calibration procedures are used (page 8000B-16, section 7.4.2.1), then calibration factors must be used.

ACTION: If any data are missing or it cannot be determined how the laboratory calculated calibration factors or response factors, contact the lab for explanation/resubmittals. Make necessary corrections and note any problems in the data assessment.

9.5 Are there any transcription/calculation errors between raw data and data summary forms. ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and document the effect in data assessments.

9.6 Are standard retention time (RT) windows for each analyte of interest presented on modified CLP summary forms? [ ] ___ ___

YES NO N/A

ACTION: If any data are missing, or it cannot be determined how RT windows were calculated, call the lab for explanation/resubmittals. Note any problems in the data assessment.

NOTE: Retention time windows for all pesticides are established using retention times from three calibration standards analyzed during the entire analytical sequence (page 8081B-15, section 7.4.6).

A 72 hr. sequence is not required with this method, however, the method states that best results are obtained using retention times which span the entire sequence; i.e., using the mid level from the 5 pt. calibration, one of the mid-concentration standards analyzed during mid-sequence and one analyzed at the end.

9.7 Were RT windows on the confirmation column established using three standards as described above? [ ] ___ ___

NOTE: RT windows for the confirmation column should be established using a 3 pt. calibration, preferably spanning the entire analytical sequence as described in 9.6 above. If RT windows on one column are tighter than the other, this may result in false negatives when attempting to identify compounds in the samples.

ACTION: Note potential problems, if any, in the data assessment.

9.8 Do all standard retention times in each level of the initial 5 pt. calibrations for pesticides fall within the windows established during the initial calibration sequence?

[ ] ___ ___

ACTION: i. If no, all samples in the entire analytical sequence are potentially affected. Check to see if three standards, spanning the entire sequence were used to obtained RT windows. If the lab used three standards from the 5 pt., RT windows

YES NO N/A

may be too tight. If so, RT windows should be recalculated as per page 8081B-15, section 7.4.6.2

ii. Alternatively, check to see if the chromatograms contain peaks within an expanded window surrounding the expected retention times.

If no peaks are found and the surrogates are visible, non-detects are valid. If peaks are present but cannot be discerned through pattern recognition or by using revised RT windows, qualify all positive results and non-detects as unusable, "R".

ACTION: For toxaphene and chlordane, the RT may be outside the RT window, but these analytes may still be identified from their individual patterns.

9.9 Has the linearity criteria for the initial calibration standards been satisfied for both columns? (% RSD must be < allowable limits* for all analytes). [ ] ___ ___

ACTION: If no, follow the actions in Table 9 below.

Table 9. Initial Calibration Linearity Criteria

Criteria	Criteria	
	Detected Associated Compounds	**Non-Detected Associated Compounds**
% RSD exceeds allowable limits*	J	No qualification
% RSD within allowable limits*	NO qualifications	

* %RSD ≤ 20% for single component compounds except alpha-BHC and delta-BHC.
%RSD ≤ 25% for alpha-BHC and delta-BHC
%RSD ≤ 30% for Toxaphene peaks
%RSD ≤ 30% for surrogates(tetrachloro-m-xylene and decachlorobiphenyl).

9.10 Has a calibration verification standard containing all analytes of interest been analyzed on each

YES NO N/A

working day, prior to sample analyses (pages 8081B-15,sections 7.5.2)? [ ] ___ ___

9.11 Has a calibration verification standard also been analyzed after every 10 samples and at the end of each analytical sequence (page 8081B-15, section 7.5.2)? [ ] ___ ___

ACTION: If no, take action as specified in section 3.2 above.

9.12 Has no more than 12 hours elapsed from the injection of the opening CCV and the end of the analytical sequence (closing CCV). Has no more than 72 hours elapsed from the injection of the sample with a Toxaphene detection and the Toxaphene CCV? [ ] ___ ___

ACTION: See Table 10 below.

9.13 Has the percent difference (%D) exceeded ± 20% for any organochlorine pesticide analyte in any calibration verification standard? ___ [ ] ___

9.14 Has a new 5 pt. calibration curve been generated for those analytes which failed in the calibration verification standard (page 8081B-16, section 7.5.2.2), and all samples which followed the out-of-control standard (page 8081B-16, section 7.5.2.3)reinjected? [ ] ___ ___

ACTION: If the %D for any analyte exceeded the ± 20% criterion and the instrument was not recalibrated for those analytes, see table below.

9.15 Have <u>daily</u> retention time windows been properly calculated for each analyte of interest (page 8081B-16, section 7.5.3)), using RTs from the associated mid concentration standard and standard deviation from the initial calibration)? [ ] ___ ___

YES NO N/A

ACTION: If no, take action specified in section 3.2 above or recalculate RT windows using the procedure outlined in method 8081B-16, section 7.5.3.

9.16 Do all standard retention times for each mid concentration standard fall within the windows established during the initial calibration sequence? [ ] ___ ___

9.17 Do all standard retention times for each mid-concentration standard (analyzed after every 10 samples) fall within the daily RT windows (page 8081B-16, section 7.5.3)? [ ] ___ ___

ACTION: If the answer to either 9.15 or 9.16 above is no, check the chromatograms of all samples which followed the last in-control standard. All samples analyzed after the last in-control standard must be re-injected, if initial analysis indicated the presence of the specific analyte that exceeded the retention time criteria (page 8081B-18, section 7.5.7.). If samples were not re-analyzed, document under Contract Non-compliance in the Data Assessment.

Reviewer has two options to determine how to qualify questionable sample data. First option is to determine if possible peaks are present within daily retention time window. If no possible peaks are found, non-detects are valid. If possible peaks are found (or interference), qualify positive hits as presumptively present "NJ" and non-detects are rejected "R". Second option is to use the ratio of the retention time of the analyte over the retention time of either surrogate. The passing criteria is ± 0.06 RRT units of the RRT of the standard component. Reject "R" all questionable analytes exceeding criteria, and "NJ" all other positive hits.

For any multi-response analytes, retention time windows should be used but analyst and reviewer should rely primarily on pattern recognition or use option 2 specified in paragraph above.

YES NO N/A

See Table 10 below.

Table 10. CCV Criteria

Criteria	Action	
	Detected Associated Compounds	Non-Detected Associated Compounds
RT out of RT window	Use professional judgement	
%D not within +/- 20%	J	UJ
Time elapsed greater than section 9.12 criteria.	R	
%D, time elapsed, RT are all within acceptable limits.	No qualifications	

9.18 Are there any transcription/calculation errors between raw data and data summary forms? ___ [ ] ___

ACTION: If large errors exists, call lab for explanation/resubmittal, make any necessary corrections and document the effect in data assessments under "Conclusions".

10.0 Analytical Sequence Check (Form VIII-PEST/Equivalent)

10.1 Have all samples been listed on CLP Form VIII or equivalent, and are separate forms present for each column? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

10.2 Was the proper analytical sequence followed for each initial calibration and subsequent analyses? [ ] ___ ___

ACTION: If no, use professional judgement to determine the severity of the effect on the data and qualify it

YES NO N/A

accordingly. Generally, the effect is negligible unless the sequence was grossly altered or the calibration was also out of limits.

11.0 Extraction Method Cleanup Efficiency Verification (Form IX/Equivalent)

11.1 Method 8081B permits a variety of extraction techniques to be used for sample preparation. Which extraction procedure was used?

1. Aqueous samples:

   1. Separatory funnel (Method 3510)_____

   2. Continuous liquid-liquid extraction (Method 3520)_____

   3. Solid phase extraction (Method 3535)_____

   4. Other _____

2. Solid samples:

   1. Soxhlet (Method 3540) _____

   2. Automated Soxhlet (Method 3541)_____

   3. Pressurized fluid (Method 3545) _____

   4. Microwave extraction (Method 3546)_____

   5. Ultrasonic extraction (Method 3550)_____

   6. Supercritical fluid (Method 3562) _____

   7. Other _____

11.2 Is Form IX - Pest-1/Equivalent present and complete for each lot of Florisil/Cartridges used? (Florisil Cleanup, Method 3620A, is required for all organochlorine pesticide extracts.) [ ] ___ ___

YES NO N/A

ACTION: If no, take action specified in 3.2 above. If data suggests that florisil cleanup was not performed, make note in the reviewer narrative.

NOTE: Method 3620A uses Florisil, while the SOW/CLP allows for Florisil cartridges. Method 3620A does not list which pesticides and surrogate(s) to use to verify column efficiency. The reviewer must check project plan to verify method used as well as the correct pesticide list. If not stated or available, use the CLP listing or accept what the laboratory used.

11.3 Are all samples listed on modified CLP Pesticide Florisil/Cartridge Check Form? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above.

11.4 If GPC Cleanup was performed, is Form IX - Pest-2/ Equivalent present? [ ] ___ ___

ACTION: If GPC was not performed and sample results indicate significant sulfur interference, make note in the data assessment.

NOTE: GPC cleanup is not required and is optional. The reviewer should check Project Plan to verify requirement.

11.5 Were the same compounds on Form IX used to check the efficiency of the cleanup procedures? [ ] ___ ___

11.6 Are percent recoveries (% R) of the pesticide and surrogate compounds used to check the efficiency of the cleanup procedures within QC limits listed on Form IX:

80-120% for florisil cartridge check? [ ] ___ ___

80-110% for GPC calibration? [ ] ___ ___

YES NO N/A

Qualify only the analyte(s) which fail the recovery criteria as follows:

ACTION: If % R are < 80%, qualify positive results "J" and quantitation limits "UJ". Non-detects should be qualified "R" if zero %R was obtained for pesticide compounds. Qualify positive results "J" (estimated).

NOTE: If 2,4,5-trichlorophenol was used to measure the efficiency of the Florisil cleanup and the recovery was > 5%, sample data should be evaluated for potential interferences.

12.0 Pesticide Identification

12.1 Has CLP Form X, showing retention time data for positive results on the two GC columns, been completed for every sample in which a pesticide was detected? [ ] ___ ___

ACTION: If no, take action specified in 3.2 above, or compile a list comparing the retention times for all sample hits on the two columns.

12.2 Are there any transcription/calculation errors between raw data and data summary forms (initial calibration summaries, calibration verification summaries, analytical sequence summaries, GPC and Florisil cleanup verification forms)? ___ [ ] ___

ACTION: If large errors exist, call lab for explanation/resubmittal, make necessary corrections and note error in the data assessment.

12.3 Are retention times (RT) of sample compounds within the established RT windows for both analyses? [ ] ___ ___

Note: Confirmation can be supported by other qualitative techniques such as GC/MS (Method 8270), or GC/AED (Method 8085) if sensitivity permits.

YES NO N/A

ACTION: Qualify as unusable (R) all positive results which were not confirmed by second GC column analysis. Also qualify "R", unusable, all positive results not within RT windows unless associated standard compounds are similarly biased. The reviewer should use professional judgement to assign an appropriate quantitation limit.

12.4 Check chromatograms for false negatives, especially if RT windows on each column were established differently (see section 9.7 above). Also check for false negatives among the multiple peak compounds toxaphene and chlordane. Were there any false negatives? ___ [ ] ___

ACTION: Use professional judgement to decide if the compound should be reported. If there is reason to believe that peaks outside retention RT windows should be reported, make corrections to data summary forms (Form I) and note in data assessment.

12.5 Was GC/MS confirmation used as the second column Confirmation? (This is not required). [ ] ___ ___

12.6 Is the percent difference (%D) calculated for the positive sample results on the two GC columns <25.0%? [ ] ___ ___

NOTE: The method 8081B requires quantitation from one column. The second column is to confirm the presence of an analyte. Calibration for the Confirmation column is a one point calibration. It is the reviewer's responsibility to verify from the project plan what the lab was required to report. If the lab was required to report concentrations from both columns, continue with validation for % Difference. If required, but not reported, either contact the lab for results or calculate the concentrations from the calibration. If not required, skip this section. Document actions in Data Assessment.

YES NO N/A

ACTION: If the reviewer finds neither column shows interference for the positive hits, the data should be qualified as follows:

% Difference	Qualifier
0-25%	none
26-70%	"J"
71-100%	"NJ"
101-200% (No Interference)	"R"
101-200% (Interference detected) "NJ"	
>50% (Pesticide vale is <CRQL)	"U"
>201%	"R"

Note: The lower of the two values is reported on Form I. If using professional judgement,the reviewer determines that the higher result was more acceptable, the reviewer should replace the value and indicate the reason for the change in the data assessment.

13.0 Compound Quantitation and Reported Detection Limits

13.1 Are there any transcription/calculation errors in Form I results? Check at least two positive values. Were any errors found? ___ [ ] ___

NOTE: Single-peak pesticide results can be checked for rough agreement between quantitative results obtained on the two GC columns. The reviewer should use professional judgement to decide whether a much larger concentration obtained on one column versus the other indicates the presence of an interfering compound. If an interference is suspected, the lower of the two values should be reported and qualified according to section 12.6 above. This necessitates a determination of an estimated concentration on the confirmation column. The narrative should indicate that the presence of interferences has led to the quantitation of the second column confirmation results.

YES NO N/A

13.2 Are the EDLs (Estimated Detection Limits) adjusted to reflect sample dilutions and, for soils, % moisture? [ ] ___ ___

ACTION: If errors are large, call lab for explanation/resubmittal, make any necessary corrections and document effect in data assessments.

ACTION: When a sample is analyzed at more than one dilution, the lowest EDLs are used (unless a QC exceedance dictates the use of the higher EDL data from the diluted sample analysis). Replace concentrations that exceed the calibration range in the original analysis by crossing out the value on the original Form I and substituting it with data from the analysis of diluted sample. Specify which Form I is to be used, then draw a red "X" across the entire page of all Form I's that should not be used, including any in the summary package.

ACTION: EDLs affected by large, off-scale peaks should be qualified as unusable, "R". If the interference is on-scale, the reviewer can provide a modified EDL flagged "UJ" for each affected compound.

14.0 Chromatogram Quality

14.1 Were baselines stable? [ ] ___ ___

14.2 Were any electropositive displacement (negative peaks) or unusual peaks seen? ___ [ ] ___

ACTION: Note all system performance problems in the data assessment.

15.0 Field Duplicates

15.1 Were any field duplicates submitted for organochlorine pesticide analysis? [ ] ___ ___

ACTION: Compare the reported results for field duplicates and calculate the relative percent difference.

ACTION: Any gross variation between field duplicate results must be addressed in the reviewer narrative. However, if large differences exist, the identity of the field duplicates is questionable. An attempt should be made to determine the proper identification of field duplicates.

METALS

This SDG contains metals using SW-846 methods _____. Region II validation guidelines were used as applicable, however, the Region has not developed an SOP for this method so these worksheets are used as an alternative.

HOLDING TIMES

Sampling Date:
Received Date: Cooler temps OK, receipt documentation present
Prep. Dates:
Analysis Dates:

All holding time requirements *were/were not* met. Receipt documentation *was/was not* present and complete. Preparation documents *were/were not* present and complete. Preservation criteria *were/were* not met. Percent moisture values *were/were not* <50% in all samples. Qualifications need:

CALIBRATIONS

All initial calibration criteria *were/were not met* for the methods noted above. All ICP Tuning and internal standard requirements *were/were not* met. All calibration verification criteria *were/were not* met. Raw data was verified. Qualifications needed:

BLANK SUMMARY

Blank qualification guidelines:

- No action is taken if an analyte is found in the blank but not in the sample.
- Sample weight, volume or dilution factor must be taken into consideration when applying the criteria.
- See blank type specific validation guidelines noted below (taken from the Region II SOP for CLP metals and adapted to this project).
- Qualification/Action codes:

Note: non-detect results are reported to the LOD so the LOD is the RL for this project.

ICB/CCB/PB Action:

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.
U - The sample result is greater than or equal to the MDL but less than or equal to the RL, result is reported as non-detect at the reporting limit, when the ICB/CCB/PB result is less than the RL.
R - Sample result is greater than the RL and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the RL.
J - Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the RL.
J/UJ - Sample result is less than 10X RL when blank result is below the negative RL.

Field QC Blank action:

Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.
Do not use rinsate blank associated with soils to qualify water samples and vice versa.

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.
U - The sample result is greater than or equal to the MDL but less than or equal to the RL when the field blank result is greater than the RL - result is reported as non-detect at the reporting limit.
R - Sample result is greater than the RL and less than the field blank value when the field blank result is greater than the RL.
J - Sample result is greater than the field blank value but less than 10X the field blank value when field blank result is greater than the RL.

Blank Contamination and Qualification Summaries

Blank ID	Analyte	Concentration	Action Level	Q Flag

Associated samples and required qualifications are noted in the following table.

SWMU _____
SDG _____
Methods: _____

Sample ID	Analyte	Q Flag

See validation report for specific samples and qualifications. Only those analytes requiring action are listed here. Negative contamination in a prep blank or CCB, if less than the analyte CRDL, is qualified based on professional judgment. Field QC blank associations are determined using tracking provided by the client. Flags are applied to samples based on these associations. **The concentration noted for the CCBs is the highest concentration in all the associated CCBs. However, when qualifying samples for CCB contamination, associated samples are those just prior to or just following a CCB. Therefore, not all analytes in all samples are flagged for CCB contamination.**

MATRIX SPIKE/DUPLICATE SUMMARY

Qualifications were made if both spike aliquots exhibited non-compliant recoveries or if one spike aliquot exhibited a non-compliant recovery and the PDS exhibited a non-compliant recovery in the same direction. Region II flagging conventions are used.

The associated matrix spike pair analyzed in this SDG _____ exhibited *compliant/non-compliant* recoveries.

List non-compliant recoveries and note any qualifications required:

The MS/MSD RPD results *were /were not* acceptable for all analytes. List non-compliant RPDs and note any qualifications required:

All LCS results *were/were not* within laboratory QC limits for all analytes/methods. List non-compliant LCS results and note any qualifications required:

The matrix duplicate of sample _____ *did/did not* meet criteria for all analytes. List non-compliant RPDs and note any qualifications required:

SERIAL DILUTIONS

The criteria used for considering the %Ds for flagging were that the criteria of >10%D applies if the concentration in the samples was greater than 50X the LOD. The PDS was also assessed as follows: if the concentration in the samples was less than 50X the LOD and the %D was high (>10%) – if the PDS was in, results were not qualified and if the PDS was out, results were qualified for that analyte. This is based on the requirements in the DOD QSM 4.2 modified using 50X LOD rather than 50X LOQ because the RL in this project is the LOD.

The serial dilution analysis of sample _____ exhibited compliant/non-compliant %Ds. List non-compliant %Ds and note any qualifications required:

SAMPLE RESULT VERIFICATION

Specific Comments:

All sample results *were/were not* reported within the calibration/linear range of the instruments. Raw data and calculations were verified. Field duplicate reproducibility was assessed – see next page. Additional qualifications *were/were not* required.

Reviewer _____ **Date:** ___/___/___

SWMU _____
SDG _____
Methods: _____

PROJECT : _____

METALS CALCULATIONS

SDG ABC

ICP - AES	
SAMPLE ID	AXYZ
ANALYTE	TIN
Lab Value CONCENTRATION (mg/Kg)	1.63
AMOUNT FROM RAW DATA (ug/L)	15.24667
SAMPLE SIZE (g)	1.09
FINAL VOLUME (L)	0.1
DILUTION FACTOR	1
DRYNESS FACTOR	0.856
CONVERSION FACTOR (g TO Kg)	1
CALCULATED CONCENTRATION (mg/Kg)	1.63

FIMS	
SAMPLE ID	BXYZ
ANALYTE	mercury
Lab Value CONCENTRATION (mg/Kg)	0.0118
AMOUNT FROM RAW DATA (ug/L)	0.064
SAMPLE SIZE (g)	0.63
FINAL VOLUME (L)	0.1
DILUTION FACTOR	1
DRYNESS FACTOR	0.858
CONVERSION FACTOR (g TO Kg)	1
CALCULATED CONCENTRATION (mg/Kg)	0.0118

ICP-MS	
SAMPLE ID	CXYZ
ANALYTE	copper
Lab Value CONCENTRATION (mg/Kg)	78.9
AMOUNT FROM RAW DATA (ug/L)	153.09123
SAMPLE SIZE (g)	1.08
FINAL VOLUME (L)	0.5
DILUTION FACTOR	1
DRYNESS FACTOR	0.898
CONVERSION FACTOR (ug/g to mg/Kg)	1
CALCULATED CONCENTRATION (mg/Kg)	78.926

DataQual **Metals**

FIELD DUPLICATE SAMPLE SUMMARY

Sample ID: AXYL
Duplicate Sample ID: AXYLD

Analyte	Sample Conc.	Dup. Sample Conc.	%RPD
antimony			0
arsenic	0.851	0.707	0.144
barium	78	72.8	7
beryllium	0.184	0.153	0.031
cadmium	0.275	0.234	0.041
chromium	21	17.4	19
cobalt	21	17.7	17
copper	108	78.9	31
lead	4.09	3.01	30
mercury			0
nickel	11.3	9.31	19
selenium	0.413	0.371	0.042
silver	0.0712	0.0468	0.0244
thallium	0.0807	0.0511	0.0296
vanadium	117	105	11
tin			0.0000
zinc	47.8	42.7	11

COMMENTS: No qualifications
Region II QC limits and flagging conventions were applied using the LOD as the reporting limit rather than the LOQ.

WET CHEMISTRY

This SDG contains _____ analyses using method(s) _____.
Region II has not developed an SOP for this method so these worksheets are used as an alternative. Region II flagging conventions are used as applicable.

HOLDING TIMES

Sampling Date:
Received Date:
Prep. Dates:
Analysis Dates:

All holding time requirements *were/were not* met. Receipt documentation *was/was not* present and complete. Preparation documents *were/were not* present and complete. Preservation criteria *were/were* not met. Percent moisture values *were/were not* <50% in all samples. Qualifications needed:

CALIBRATIONS

All initial calibration criteria *were/were not met* for the methods noted above. All calibration verification criteria *were/were not* met. Raw data was verified. Qualifications needed:

BLANK SUMMARY

Blank qualification guidelines:

- No action is taken if an analyte is found in the blank but not in the sample.
- Sample weight, volume or dilution factor must be taken into consideration when applying the criteria.
- See blank type specific validation guidelines noted below (taken from the Region II SOP for CLP metals and adapted to this project).
- Qualification/Action codes:

Note: non-detect results are reported to the LOD so the LOD is the RL for this project.

ICB/CCB/PB Action:

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.
U - The sample result is greater than or equal to the MDL but less than or equal to the RL, result is reported as non-detect at the reporting limit, when the ICB/CCB/PB result is less than the RL.
R - Sample result is greater than the RL and less than the ICB/CCB/PB value when the ICB/CCB/PB value is greater than the RL.
J - Sample result is greater than the ICB/CCB/PB value but less than 10X the ICB/CCB/PB value when ICB/CCB/PB value is greater than the RL.
J/UJ - Sample result is less than 10X RL when blank result is below the negative RL.

Field QC Blank action:

Note – Use field blanks to qualify data only if field blank results are greater than prep blank results.
Do not use rinsate blank associated with soils to qualify water samples and vice versa.

No Action - The sample result is greater than the RL and greater than ten times (10X) the blank value.
U - The sample result is greater than or equal to the MDL but less than or equal to the RL when the field blank result is greater than the RL - result is reported as non-detect at the reporting limit.
R - Sample result is greater than the RL and less than the field blank value when the field blank result is greater than the RL.
J - Sample result is greater than the field blank value but less than 10X the field blank value when field blank result is greater than the RL.

Blank Contamination and Qualification Summaries

Blank ID	Analyte	Concentration	Action Level	Q Flag

Associated samples and required qualifications are noted in the following table.

Sample ID	Analyte	Q Flag

See validation report for specific samples and qualifications. Only those analytes requiring action are listed here. Negative contamination in a prep blank or CCB, if less than the analyte CRDL, is qualified based on professional judgment. Field QC blank associations are determined using tracking provided by the client. Flags are applied to samples based on these associations. **The concentration noted for the CCBs is the highest concentration in all the associated CCBs. However, when qualifying samples for CCB contamination, associated samples are those just prior to or just following a CCB. Therefore, not all analytes in all samples are flagged for CCB contamination.**

MATRIX SPIKE/DUPLICATE SUMMARY

Qualifications were made if both spike aliquots exhibited non-compliant recoveries or if one spike aliquot exhibited a non-compliant recovery and, if a PDS was performed, it exhibited a non-compliant recovery in the same direction. Region II flagging conventions are used.

The associated matrix spike pair analyzed in this SDG _____ exhibited *compliant/non-compliant* recoveries.

List non-compliant recoveries and note any qualifications required:

The MS/MSD RPD results *were /were not* acceptable for all analytes. List non-compliant RPDs and note any qualifications required:

All LCS results *were/were not* within laboratory QC limits for all analytes/methods. List non-compliant LCS results and note any qualifications required:

The matrix duplicate of sample _____ *did/did not* meet criteria for all analytes. List non-compliant RPDs and note any qualifications required:

SAMPLE CALCULATION

EPA SAMPLE ID: XABC
Parameter: TOC
Concentration: 1200 mg/L
Dilution Factor: 20
%Solids – na

Raw Data result: 60.287 mg/L * 20 = 1205.74 mg/L

FIELD DUPLICATE SAMPLE SUMMARY

Note: In general, region II guidance for metals field duplicates are applied to these wet chemistry method as applicable.

Sample ID: none Duplicate Sample ID:

Comments: No qualifications were required

SAMPLE RESULT VERIFICATION

Specific Comments:
All sample results *were/were not* reported within the calibration/linear range of the instruments. Raw data and calculations were verified. Additional qualifications *were/were not* required.

Reviewer _____ **Date:** ___/___/___

SWMU _____
SDG _____
Methods: _____

APPENDIX F
SAMPLE LABEL AND CUSTODY DOCUMENTATION

Project Name: _____ Project No: _____

Sample ID: _____

Sample Date: _____ Sample Time: _____

Sampler(s): _____

Analyses: _____

Preservatives: _____

Environmental Testing Laboratories

CompuChem

A Division Of

Liberty Analytical Corp.

CUSTODY SEAL

Signature

Date

S 13612

SUBCONTRACT CHAIN-OF-CUSTODY RECORD

COMPUCHEM
a division of Liberty Analytical Corp.

501 Madison Avenue
Cary, NC 27513
1-800-833-5097

Project Name:	Samples shipped to:	CompuChem point-of-contact: (4)
	Contact:	
TAT:	Address:	Phone: (919) 379-4100 X_____
		Fax: (919) 379-_____
Report style:		
	Phone: ( )	Sampling complete? Y or N (see Note 1)
Disk requirement:	Project Locale (state)	Project-specific (PS) or Batch (B) QC? _____

BOX #1	BOX #2	BOX #3	BOX #4	BOX #5
1. Surface Water 2. Ground Water 3. Leachate 4. Rinsate 5. Soil / Sediment / Sludge 6. Trip Blank 7. Oil 8. Waste 9. Other _____	A. HCl + Ice B. HNO_3 + Ice C. NaOH + Ice D. H_2SO_4 + Ice E. Unpreserved F. Ice Only G. Other _____ H. $NaHSO_4$ + Ice I. ZnAc+NaOH + Ice J. Methanol	F- Filtered U- Unfiltered	H- High M- Medium L- Low	C- CLP T- TCLP S - SW-846 W- CWA 600-series O- Other _____

Sample ID	Date / Year	Time	Box #1 Matrix	Box #2 Preservative	Box #3 Filtered / Unfiltered	Box #4 Expected Conc.	Box #5 Method	# of Bottles	Use for Lab QC (MS or DUP)	PARAMETERS					CCN	Remarks / Comments (see Notes 2 & 3)

Clients Special Instructions: Temperature _____°C

Lab: Received in good condition? Y or N Describe any problems:

#1 Relenquished by:(sig)	Date:	#2 Relinquished by:(sig)	Date:	Relinquished by:(sig)	Date:
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:
#1 Received by:(sig)	Date:	#2 Received by:(sig)	Date:	#2 Received by:(sig)	Date:
Company Name:	Time:	Company Name:	Time:	Company Name:	Time:

Note (1) If "N" lab should batch samples to await remainder of project - maximizing batch size and minimizing QC ratio; if "Y" lab should begin processing batches now.
Note (2) Samples should be stored 60 days after date report mailed at no extra charge.
Note (3) All lab copies of data should be retained for a minimum of 3 years.
Note (4) Please call point-of-contact to verify receipt of samples.

Revision 0 2-10-99

APPENDIX G
LABORATORY EDD REQUIREMENTS

Baker Laboratory EDD

Michael Baker Jr, Inc
Division of Michael Baker Corporation

September 2003 (revised July 2011)

Baker Laboratory EDD

For all new data sources, an electronic data deliverable (EDD) format has been established for all of Baker's analytical subcontractors. The EDD is a modified L2 format for the Terrabase Data Management System from Integrate, Inc. of Baton Rouge, LA. This structure has been chosen not only because of Baker use of the Terrabase system, but because it can be easily manipulated for use to meet Baker's internal data needs. If you have any technical questions, please contact Shannon Raub at 412-375-3043.

The analytical subcontractor's contractual requirements for the completion of any analytical services are not deemed to be met for any group of analyses performed until the Data Manager has received acceptable EDD files and description documents for all associated data as described within this guidance document and the Project QAPP. In the event of review, the EDD must, at minimum, allow the Data Manager to produce a complete and correct facsimile of the analytical subcontractor's hard-copy report for the analyses. Failure of the analytical subcontractor to comply with reasonable change requests made by the Data Manager in order to obtain complete and compliant EDD file(s) will result in rejection of the EDD and a request for resubmission of the EDD.

Structure/Definitions

All EDD data must be presented as a bar-delimited (|) text file within the strict limitations of the format described in the following table. However, even if the formatting of the EDD is completely correct, the Data Manager may choose to reject the EDD if the contents of that file do not comply with the data-library standardization requirements detailed within the attached Appendices and data libraries.

Additionally, for special projects and uses, a Data Manager responsible for data standardization may provide additions to the standard data libraries for the analytical subcontractor's special use within those projects. Any such additions shall be provided under separate cover and do not take the place of this generic data standards document.

The formatted EDD as submitted by the analytical subcontractor to the Data Manager must always be accompanied by a separate, descriptive text file, which details (at minimum):

1. The name of the associated EDD file
2. The Facility to which the data should be associated.
3. All additional information that a lab has included in any hard-copy case narrative documentation, which accompanies the associated hard-copy report.
4. A certification that the data contained in the associated EDD is an exact electronic representation for all applicable fields presented upon the associated hard-copy report.
5. The contact information for the lab or any other firms that produced the EDD file for the lab.

Typically each EDD file and its companion description file are to be compressed together into a standard ZIP file format mutually agreed upon in advance by the analytical subcontractor and the Data Manager. This ZIP file may also be encrypted and/or password protected (at the discretion of the Data Manager) in order to prevent unauthorized access to the resident data and description. The ZIP file is then electronically transmitted (using secure e-mail, bbs, ftp, etc. as directed) to the Data Manager for review and acceptance.

The TerraBase® Environmental Data Management System stores analytical data from separate geographic facilities in segregated regions within the database structure. For this reason, sample data from separate facilities can never be combined within a single EDD file. However, data from multiple Projects and

Sample Delivery Groups (SDGs) performed at the *same Lab* for the *same Facility* may (at the discretion of the Data Manager) is included within the same EDD file.

Field Number-Name	Data Type: Length	Description
1-Laboratory ID	Text: 6	Laboratory's facility identifier (assigned by Data Manager).
2-Project ID	Long Integer	Major divisions (or projects) of historical data. The data manager assigns the project numbers.
3-SDG ID	Text: 8	Unique Subdivisions of a Project ID (Sample Delivery Group) analyzed by a single Lab. Different labs may not identify their results with the same Batch ID or their EDD files will wholly or partially overwrite each other during successive TerraBase data import operations. If left blank, this will be assumed to be the year/quarter in which the sample was taken or the year/quarter the analysis was run if sample d/t is unavailable. Otherwise assigned by the Data Manager
4-Analytical Fraction	Text: 1	A logical grouping of analytical results dictated by analytical methods employed for analysis. Other Fractions cannot be added. This list is limited to the items shown above.
5-Site Sample ID	Text: 25	The sample name exactly as it appears on the associated chain of custody.
6-Sampling Date/Time	Date/Time	Date sample was collected in the format "mm/dd/yyyy hh:mm" (i.e., "05/04/1997 13:27") If no time component is applicable or known, "00:00" should be used.
7-Top Depth	Real	Top depth at which sample was taken (groundwater/soil)
8-Middle Depth	Real	Middle depth at which sample was taken (groundwater/soil)
9-Bottom Depth	Real	Bottom depth at which sample was taken (groundwater/soil)
10-Sample Point ID	Text: 20	Georeferenced data point from which a sample was taken. This field is required for data that is to be modeled, but may be blank for QC data. This entry is often similar to (if not identical to) the "Site Sample ID" field noted above. However, *it must have consistent nomenclature over repetitive sampling events* since it represents the name of a physical location on a facility map (such as using "MW-01" to always represent the "Monitor Well #01" map location).
11-Lab Sample ID	Text: 15	Sample name as assigned by laboratory/LIMS.
12-Lab Sample Type	Text: 5	Type of sample such as site sample or field duplicate. Refer to Appendix A for a complete list of supported sample types. *This list is maintained by the Data Manager in a user-editable table
13-Matrix	Text: 1	"S" = Soil/Sediment, "W" = Surface/Ground Water or Aqueous, "A" = Air, "O" = Oil, "T" = Tissue, "L" = Leachate
14-Field Sample Classification	Text: 3	Type of sample such as surface soil or groundwater. Refer to Appendix A for a complete list of supported sample types. *This list is maintained by the Data Manager in a user-editable table
15-Filtration Method	Text: 1	Filtration method where applicable (applies especially to metals and some conventional parameters): "U" = Unfiltered (Total) "F" = Filtered (Dissolved) "L"= any TCLP extraction procedures Other codes may be applied at the direction of the Data Manager
16-Extraction Date/Time	Date/Time	Applies mainly to Pesticides/PCB's and Semivolatiles. Reported in same format as Sampling Date/Time (mm/dd/yyyy hh:mm) If

		no time component is applicable or known, “00:00” should be used.
17-Preparation Date/Time	Date/Time	Applies mainly to Metals. Reported in same format as Sampling Date/Time (mm/dd/yyyy hh:mm) If no time component is applicable or known, “00:00” should be used.
18-Analysis Date/Time	Date/Time	Date sample was analyzed in the format "mm/dd/yyyy hh:mm". If no time component is applicable or known, “00:00” should be used.
19-Instrument ID	Text: 10	Laboratory instrument, which analyzed the sample.
20-Rough Percent Moisture	Integer	Percent moisture found expressed as an integer. This field should be left blank for all aqueous samples and does not reflect the Quantitative result of a “Karl-Fischer” or other “percent moisture” parameter test. The data in this field only serves to alert the Project Manager to potential calculation and correction issues that may need to be elsewhere applied during data review and reporting operations, which take place outside the Lab.
21-Dilution Factor	Real	Any Dilution Factor that which was applied to the analytical result acquired upon the analytical testing instrument in order to produce the final result reported upon the lab’s hard-copy report for this analyte. As such, it does not include prep-related dilutions for analytical operations. At the discretion of the Data Manager, sample preps and their associated dilutions which affect and entire sample may also be reportable in the same EDD with the prep method listed as the “Analytical Method”
22-Analyte Type	Text: 1	"A" = Analyte, "T" = Tentatively Identified Compound (TIC-for Organic analyses only), “I” = Internal Standard, “S” = Surrogate Compound No other values are acceptable
23-Analytical Method	Text: 13	Standardized code representing the approved Analytical method used in instrumental or classical chemistry evaluation, which produced the final result for the reported parameter (e.g.: 150.1, SM9222B, 8260B, etc.).
24-CAS Number Equivalent	Text: 9	Chemical Abstract Service Number *without hyphens* where applicable (i.e., "000012345" will equate to "000012-34-5"). If the parameter is not actually a chemical at all (e.g.: “pH” or “Conductivity”) the lab should obtain a CAS-Number-Equivalent from the Data Manager for this field or leave it blank. CAS numbers are the most critical index for all analytical result parameters. However, compounds that are designated to be Organic TICs may be reported without CAS values in this field.
25-Parameter Name	Text: 67	The assigned name of the compound or parameter being tracked. The Analyte Name *must* be reported consistently on all deliverables (especially for TICs, when CAS Numbers are not provided).
26-Retention Time	Text: 5	Required for TICs in the format mm:hh (minutes and hundredths of a minute) in lieu of CAS values.
27-Detection/Reporting Limit	Text: 11	The detection/reporting limit must be reported in the same Result Units as an analyte’s Quantitative Result, and should only be corrected for any applied analytical dilutions. Otherwise this value should reflect only the routine, undiluted detection/reporting limit for each analyte. The detection limit threshold is typically based upon one or

		more of the following: 1. The instrument detection limit (IDL) for the analytical method utilized (where applicable). The associated method detection limit (MDL), 2. The practical quantitation limits (PQL), 3. The contract required detection/reporting limits (such as the CRDL and CRQL commonly used for CLP procedures).
28-MDL	Text: 11	Method Detection Limit (Baker added field)
29-Laboratory Quantitative Result	Text: 11	Concentrations or other numerical, quantifiable, readings such as pH or temperature. If a value is non-detect, please put the limit of detection value in this field. Symbols such as “ND” “<” or “>” are not acceptable in this field. For any analytical methods which properly produce a “text” result (such as “Color”), or for those which cannot produce a precise numerical value (such as “Total Coliform” which can be “Too Numerous to Count”), this field must be left ***empty*** (i.e., “null”) and the text-value or an acceptable abbreviation (not to exceed five characters) must be placed in the associated “Lab Qualifier” field
30-Laboratory Qualifier	Text: 5	The analytical subcontractor must supply the qualifiers. For short, “Text” results (as described above) please obtain guidance from the Data Manager to secure standardization of abbreviations, etc. If the value was non-detect in the laboratory quantitative result column, please place a "U" in this field.
31-Result Units	Text: 8	Examples of typical Result Units (others may also be used as appropriate if directed by the Project Manager): 1. mg/L, mg/Kg, or PPM for various analytes 2. ug/L, ug/Kg, or ppb for various analytes 3. mg/m3, ppmv or ppbv for air analyses 4. ug/g for tissue analyses 5. S.U. (standard units) for pH 6. Umhos/cm for specific conductivity 7. C (centigrade) or F (Fahrenheit) for flashpoint 8. % for lower explosive limit (LEL) 9. pCi/L for radiologicals
32-LOD	Text: 11	Limit of Detection (Baker added field)

Appendix A

Standard TerraBase® codes for use with aforementioned EDDs and reports:

This section is provided for engineering and laboratory staff reference when constructing hard copy reports and EDDs in the aforementioned applicable formats for data submission to the Data Manager. Please contact Baker staff directly with any additional questions related to these standards and codes.

1. Lab Sample Types (Field Number 12)

Lab Sample Type (Code)	Lab Sample Description	Appropriate Field Sample ID Suffix (when applicable)
BS	Blank Spike	
BSD	Blank Spike Duplicate	
BSDRE	Blank Spike Duplicate Re-extraction/Re-analysis	RE
BSRE	Blank Spike Re-extraction/Re-analysis	RE
CC	Continuing Calibration	
CV	Calibration Verification	
DL	Dilution	DL
ER	Equipment Rinsate	
ERDL	Equipment Rinsate Dilution	DL
ERRE	Equipment Rinsate Re-extraction/Re-analysis	RE
FB	Field Blank	
FBRE	Field Blank Re-extraction/Re-analysis	RE
FD	Field Duplicate	
FDDL	Field Duplicate Dilution	DL
FDRE	Field Duplicate Re-extraction/Re-analysis	RE
FLB	Leachate Fluid Blank	
FLO	Florisil Cartridge Check	
IB	Instrument Blank	
IC	Initial Calibration	
IPC	Instrument Performance Check	
LCS	Lab Control Sample	
LCSRE	Lab Control Sample Re-extraction/Re-analysis	RE
LD	Lab Duplicate	
LRB	Laboratory Reagent Blank	
MB	Method Blank	
MBRE	Method Blank Re-extraction/Re-analysis	RE
MS	Matrix Spike	
MSD	Matrix Spike Duplicate	
MSDDL	Matrix Spike Duplicate Dilution	DL
MSDL	Matrix Spike Dilution	DL
MSDRE	Matrix Spike Duplicate Re-extraction/Re-analysis	RE
MSRE	Matrix Spike Re-extraction/Re-analysis	RE
SB	Storage Blank	
SBRE	Storage Blank Re-extraction/Re-analysis	RE

TB	Trip Blank	
TBRE	Trip Blank Re-extraction/Re-analysis	RE
TRG	Site Sample	
TRGDL	Site Sample Dilution	DL
TRGRE	Site Sample Re-extraction/Re-analysis	RE
TRGRS	Site Sample Re-sampling	

2. Field Sample Classifications (EDD Field Number 14):

Lab Sample Classification ID (Code)	Lab Sample Classification Description
AAS	Air
BW	Bottom of Column Water
MW	Mid-Column Water
QA	Quality Assurance/Quality Control
SE	Sediment
SO	Soil, unidentified
SS	Surface Soil
SU	Subsurface Soil
SW	Surface Water / Top of Column
TC	Crawfish Tissue
TCR	Crab Tissue
TF	Fish Tissue
TM	Mussel Tissue
TO	Oyster Tissue
TS	Shrimp Tissue
TW	Groundwater

3. Laboratory Analytical Fraction Identifiers (EDD Field Number 4):

Fraction (Code)	Fraction Name
A	PAHs
B	Semivolatiles
C	Conventional (Wet Chemistry, default)
D	Dissolved Metals
E	Explosives
F	Dioxins/Furans
G	PCBs (Stand Alone Analysis)
H	Herbicides
I	Dissolved Gases
M	Metals
N	Natural Attenuation Parameters
O	Orthophosphates Pesticides
P	Pesticides/PCBs
Q	TCLP Analysis (Both Organic and Inorganic)
R	Radiological
T	Total Petroleum Hydrocarbons

V	Volatile
X	BTEX

APPENDIX H
ECOLOGICAL RECEPTORS AND SPECIES OF SPECIAL CONCERN

APPENDIX H

ECOLOGICAL RECEPTORS AND SPECIES OF SPECIAL CONCERN
SWMU 60 – FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

A description of the fauna (including species of special concern) that may potentially reside and/or forage within SWMU 60 and adjacent habitats are identified and discussed below.

Mammals

A total of twenty-two terrestrial mammal species are known historically from Puerto Rico; however, all mammals except bats (thirteen species) have been extirpated (Mac et al., 1998). None of the bats found on Puerto Rico are exclusive to the island, nor are they listed under provisions of the Endangered Species Act of 1973. The specific bat species known to occur on Puerto Rico are listed below:

- Fruit-eating bats: Jamaican fruit bat (*Artibeus jamaicensis*), Antillean fruit bat (*Brachyphylla cavernarum*), and red fig-eating bat (*Stenoderma rufum*)

- Nectivorous bats: brown flower bat (*Erophylla sezekoni*) and greater Antillean long-tounged bat (*Monophyllus redmani*)

- Insectivorous bats: Antillean ghost-faced bat (*Mormoops blainvillii*), Parnell's mustached bat (*Pteronotus parnellii*), sooty mustached bat (*Pteronotus quadridens*), big brown bat (*Eptesicus fuscus*), red bat (*Lasiurus borealis*), velvety free-tailed bat (*Molossus molossus*), and Brazilian free-tailed bat (*Tadarida brasiliensis*)

- Piscivorous bats: Mexican bulldog bat (*Noctilio leporinus*)

Vegetation growing within the undeveloped upland portion of SWMU 60 is dominated by white lead tree (*Leucaena leucocephala*). White lead tree is known to be used as a source of food by bats on Puerto Rico (nectar/pollen; Gannon et al., 2005). Several terrestrial mammals have been introduced into Puerto Rico, including the black rat (*Rattus rattus*), Norway rat (*Rattus norvegicus*), and small Indian mongoose (*Herpestes javanicus*). These nonindigenous mammals have been implicated in the decline of native bird and reptile populations (Mac et al., 1998 and U.S. Fish and Wildlife Service [USFWS], 1996a).

Of the endangered/threatened marine mammals that may occur in Puerto Rico, only the West Indian manatee is known to occur in the marine environment surrounding NAPR (DoN, 2007). The turtle grass climax meadows of the Ensenada Honda represent grazing areas for this marine mammal. It is noted that feeding manatees at NAPR are most often observed within Pelican Cove and the Ensenada Honda.

Birds

A total of 239 bird species are native to Puerto Rico (Raffaele, 1989). This total includes breeding permanent residents and non-breeding migrants. In addition, many nonindigenous bird species have been introduced to Puerto Rico, including the shiny cowbird (*Molothrus bonariensis*) and several parrot species, such as the budgerigar (*Melopsittacus undulates*), orange-fronted parrot (*Aratinga canicularis*), and monk parrot (*Myiopsitta monaqchus*). Of the 239 species native to Puerto Rico, twelve are endemic to the island (Raffaele, 1989).

Numerous native and migratory bird species have been reported at NAPR (Geo-Marine, Inc., 1998). A list of bird species reported at NAPR or having the potential to occur is provided in Table H-1. The list, compiled from literature-based information pre-dating 1990, includes the great blue heron (*Ardea herodias*), snowy egret (*Egretta thula*), little blue heron (*Florida caerulea*), black-crowned night heron (*Nycticorax nycticorax*), belted kingfisher (*Ceryle alcyon*), spotted sandpiper (*Actitis macularia*), greater yellowlegs (*Tringa melanoleauca*), black-bellied plover (*Squatarola squatarola*), clapper rail (*Rallus longirostris*), Royal tern (*Thalasseus maximus*), sandwich tern (*Thalasseus sandvicensis*), least tern (*Stema albifrons*), yellow warbler (*Dendroica petechia*), palm warbler (*Dendroica palmarum*), prairie warbler (*Dendroica discolar*), magnolia warbler (*Dendrocia magnolia*), mourning dove (*Zenaida macroura*), red-legged thrush (*Mimocichla plumbea*), common nighthawk (*Chordeiles minor*), and red-tailed hawk (*Buteo jamaicensis*). Endemic species reported from NAPR include the Puerto Rican lizard cuckoo (*Saurothera vieilloti*), Puerto Rican flycatcher (*Myiarchus antillarum*), Puerto Rican woodpecker (*Malanerpes portoricensis*), Puerto Rican emerald (*Chlorostilbon maugaeus*), and yellow-shouldered blackbird (*Agelaius xanthomus*).

The yellow-shouldered blackbird is a federally endangered species. One of the principal reasons for the status of this species is attributed to parasitism by the nonindigenous shiny cowbird, which lays its eggs in blackbird nests and sometimes punctures the host's eggs (USFWS, 1983). Other factors contributing to the status of this species include nest predation by the introduced black rat, Norway rat, and mongoose, as well as habitat modification and destruction (USFWS, 1996a). The entire land area of NAPR was declared critical habitat for the yellow-shouldered blackbird in 1976; however, a 1980 agreement between the Navy and the USFWS exempted certain areas from this categorization (Geo-Marine, Inc., 1998). A study conducted by the Naval Facilities Engineering Service Center (NFESC, 1996) reported that the mangrove forests surrounding NAPR, including the E2SS3 wetland at SWMU 60, should be considered the most important nesting habitats for the yellow-shouldered blackbird. Yellow-shouldered blackbirds are arboreal feeders that forage within the canopy and sub-canopy of trees (USFWS, 1996a). Therefore, undeveloped areas of the SWMU also represent potential foraging habitat for the yellow-shouldered blackbird.

Other federally listed bird species that have the potential to occur at NAPR are the piping plover (*Charadrius melodus*) and roseate tern (*Sterna dougallii*) (Geo-Marine, Inc., 1998). The piping plover is a rare, non-breeding winter visitor in Puerto Rico (Raffaele, 1989). This species breeds only in North America in three geographic regions (Atlantic Coast population [threatened], Great Lakes population [endangered], and Northern Great Plains population [threatened]; USFWS, 1996b). No piping plover observations were reported at NAPR during the 1990s or during sea turtle nesting surveys conducted in 2002 and 2004 (Geo-Marine, Inc., 2005). No historic evidence is available to indicate whether the roseate tern (threatened in Puerto Rico) has ever nested at NAPR and no roseate tern observations have been noted in or over coastal waters adjacent to NAPR (DoN, 2007). The nearest active roseate tern colony likely occurs on the eastern end of Vieques Island (more than twenty miles east of NAPR) (DoN, 2007).

Reptiles and Amphibians

A total of twenty-three amphibians and forty-seven reptiles are known from Puerto Rico and the adjacent waters (Mac et al., 1998). Fifteen of the amphibians and twenty-nine of the reptiles are endemic, while four amphibian species and three reptilian species have been introduced (Mac et al. 1998). Puerto Rico's native amphibian species include sixteen species of tiny frogs commonly called coquis. On the coastal lowlands, almost all coqui species are arboreal. The only amphibians listed under provisions of the Endangered Species Act of 1973 are the Puerto Rican crested toad (*Peltophryene lemur*) and the golden coqui (*Eleutherodactylus jasperi*). Both species are listed as threatened (USFWS, 2012). Distribution of the golden coqui is restricted to areas of dense bromeliad growth. All specimens to date have been collected from a small semicircular area of a 6-mile radius south of Cayey (approximately 30 miles

southwest of NAPR), generally at elevations above 700 meters (USFWS, 1984). The Puerto Rican crested toad occurs at low elevations (below 200 meters) where there is exposed limestone or porous, well drained soil offering an abundance of fissures and cavities (USFWS, 1987). A single large population is known to exist from the southwest coast in Guánica Commonwealth Forest, and a small population is believed to survive on the north coast near Quebradillas, Arecibo, Barceloneta, Vega Baja, and Bayamón (USFWS, 1987). It also has been collected on the southeastern coastal plain near Coamo (USFWS, 1987). Given the habitat preferences and locations of known occurrences, these two amphibian species are not expected to occur at NAPR.

Puerto Rico's native reptilian species include thirty-one lizards, eight snakes, one freshwater turtle, and five sea turtles (Mac et al., 1998). Of the five sea turtles, only the green sea turtle, hawksbill sea turtle (*Eretmochelys imbricata*), and loggerhead sea turtle (*Dermochelys coriacea*) nest within Puerto Rico. These three sea turtles, as well as the leatherback sea turtle (*Caretta caretta)* are listed under the provisions of the Endangered Species Act of 1973 (hawksbill sea turtle and leatherback sea turtle are listed as endangered, while the green sea turtle [Caribbean population] and loggerhead sea turtle are listed as threatened; USFWS, 2011). Aerial surveys of turtles were performed from March 1984 through March 1995 along the Puerto Rican Coast. This information is summarized in the Draft NAPR Disposal Environmental Assessment (Geo-Marine, Inc., 2005). Significant turtle observations were made near the mouth of the Ensenada Honda, the northern shore of Pineros Island, Pelican Bay, and the Medio Mundo Passage, with the frequency of turtle observations listed as green > hawksbill > loggerhead > leatherback. Based on the life history information for each turtle species (summarized in Baker, 2006a and 2006b) and the availability of forage material (in the form of sea grasses), the green sea turtle has the potential to forage within the Ensenada Honda.

The Puerto Rican boa (*Epicrates inornatus*) is a federally endangered species. Four Puerto Rican boa sightings were reported at NAPR prior to 1999 and an additional four occurrences were reported between 2001 and 2003 (Geo-Marine, Inc., 2005). However, no boas were observed during 211 man-hours of surveys conducted within potential boa habitat in 2004 (Tolson, 2004). The Puerto Rican boa uses a variety of habitats but is most commonly found in Karst forest habitat (forested limestone hills) (USFWS, 1986). Based on the absence of preferred habitat, there is low probability of occurrence of this species at SWMU 60 and adjacent habitats.

Fish and Aquatic Invertebrates

A diverse fish and invertebrate community can be found in the marine environment surrounding NAPR. This can be attributed to the varied habitats that include open water marine and estuarine habitat, mud flats, sea grass beds, and mangrove forests. The fish community is represented by stingrays, herrings, groupers, needlefish, mullets, barracudas, jacks, snappers, grunts, snooks, lizardfishes, parrotfishes, gobies, filefishes, wrasses, damselfishes, and butterflyfish (Geo-Marine, Inc., 1998). The benthic invertebrate community includes sponges, corals, anemones, sea cucumbers, sea stars, urchins, and crabs.

The specific species inhabiting the Ensenada Honda have not been documented in the literature or during previous investigations. However, a marine reconnaissance survey was conducted within a small embayment associated with SWMU 45 in May 2000 [(Geo-Marine, Inc., 2000)]. Marine invertebrates observed within this embayment included sea urchins (*Echinometra lucunter* and *Echinometra viridis*), encrusting fire coral (*Millipora alcicormus*), common sea fan (*Gorgonia venalina*), starlet coral (*Siderastrea ammulatta*), pincushin starfish (*Oreaster reticulate*s), and corkscrew anemone (*Bartholomea annulatta*), as well as two species of sea cucumbers (*Actinopyga agassizii* and *Holothuria mexicana*). In addition to invertebrates, sixteen fish species were observed within the cove. The specific species encountered included sergeant major (*Abudefduf saxatillis*), dusky damselfish (*Stegates fuscus*), tomtate (*Haemulon aurolineatum*), gray snapper (*Lutjanus griseus*), squirrelfish (*Holocentrus sp*.), yellow fin

mojarra (*Gerres cinereus*), and silver jenny (*Eucinostomus gula*). Many of the species encountered during the marine reconnaissance survey are likely present within the Ensenada Honda.

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TABLE H-1

LIST OF BIRDS REPORTED FROM OR HAVING THE POTENTIAL TO OCCUR AT NAVAL ACTIVITY PUERTO RICO
SWMU 60 – FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Common Name [(1)]		
Pied-billed grebe	Red-billed tropicbird	Brown pelican
Brown booby	Magnificent frigatebird	Great blue heron
Louisiana heron	Snowy egret	Great egret
Striated heron	Little blue heron	Cattle egret
Least bittern	Yellow-crowned night heron	Black-crowned night heron
White-cheeked pintail	Blue-winged teal	American widgeon
Red-tailed hawk	Osprey	Merlin
Clapper rail	American coot	Caribbean coot
Common gallinule	Piping plover [(3)(4)]	Semipalmated plover
Black-bellied plover	Wilson's plover	Killdeer
Ruddy turnstone	Black-necked stilt	Whimbrel
Spotted sandpiper	Semipalmated sandpiper	Short-billed dowitcher
Greater yellowlegs	Lesser yellowlegs	Willet
Stilt sandpiper	Pectoral sandpiper	Laughing gull
Royal tern	Sandwich tern	Bridled tern
Least tern	Brown noddy	White-winged dove
Zenaida dove	White-crowned pigeon	Mourning dove
Red-necked pigeon	Common ground dove	Bridled quail dove
Ruddy quail dove	Caribbean parakeet	Smooth-billed ani
Yellow-billed cuckoo	Mangrove cuckoo	Short-eared owl
Chuck-will's-widow	Common nighthawk	Antillean crested hummingbird
Green-throated carib	Antillean mango	Belted kingfisher

TABLE H-1

LIST OF BIRDS REPORTED FROM OR HAVING THE POTENTIAL TO OCCUR AT NAVAL ACTIVITY PUERTO RICO
SWMU 60 – FORMER LANDFILL AT THE MARINA
FULL RFI SAMPLING AND ANALYSIS PLAN
NAVAL ACTIVITY PUERTO RICO, CEIBA, PUERTO RICO

Common Name [1]		
Gray kingbird	Loggerhead kingbird	Stolid flycatcher
Caribbean elaenia	Purple martin	Cave swallow
Barn swallow	Northern mockingbird	Pearly-eyed thrasher
Red-legged thrush	Black-whiskered vireo	American redstart
Parula warbler	Prairie warbler	Yellow warbler
Magnolia warbler	Cape May warbler	Black-throated blue warbler
Adelaide's warbler	Palm warbler	Black and white warbler
Ovenbird	Northern water thrush	Bananaquit
Striped-headed tanager	Shiny cowbird	Black-cowled oriole
Greater Antillean grackle	Yellow-shouldered blackbird [2]	Hooded manakin
Yellow-faced grassquit	Black-faced grassquit	Least sandpiper
Western sandpiper	Puerto Rican woodpecker	Rock dove
Puerto Rican emerald	Puerto Rican flycatcher	Pin-tailed whydah
Spice finch	Ruddy duck	Peregrine falcon
Marbled godwit	Puerto Rican lizard cuckoo	Prothonotary warbler
Green-winged teal	Orange-cheeked waxbill	Roseate tern [3][4]
Least grebe	West Indian whistling duck	Puerto Rican screech owl
Puerto Rican tody	Green heron	

Notes:

[1] List of birds taken from Geo-Marine, Inc. (1998).
[2] Federally-designated endangered species.
[3] Federally-designated threatened species.
[4] Species has the potential to occur at Naval Activity Puerto Rico.