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

IN REPLY REFER TO:
5090/S4.7.3
Ser 0592/8230
28 AUG 2008
8-1-08

U.S. Environmental Protection Agency, Region V
Waste, Pesticides, & Toxics Division
Waste Management Branch
Corrective Action Section LU-9J
77 West Jackson Blvd.
Chicago, IL 60604

Dear Mr. Ramanauskas:

Crane Division, Naval Surface Warfare Center submits the Final Quality Assurance Project Plan (QAPP) Addendum No. 4 for the PCB Capacitor Burial-Pole Yard, Solid Waste Management Unit (SWMU) 17. This QAPP Addendum is for sediment sampling in Boggs Creek to support the interim measures removal at SWMU 17. One copy is presented as enclosure (1). The permit required Certification Statement is provided as enclosure (2).

If you require any further information, my point of contact is Mr. Thomas J. Brent, Code 0592-TB, at 812-854-6160, email thomas.brent@navy.mil.

Sincerely,

J. M. Hunsicker
J. M. HUNSICKER
Environmental Protection Mgr
By direction of the Commanding Officer

Enclosures: 1. Final SWMU 17 QAPP Addendum No. 4
2. Certification Statement

Copy to:
ADMINISTRATIVE RECORD
NAVFAC MW (Howard Hickey) (w/o encl)
IDEM (Doug Griffin)
TTNUS (Ralph Basinski) (w/o encl)

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

James M. Hanauka

SIGNATURE

28 AUG 2008

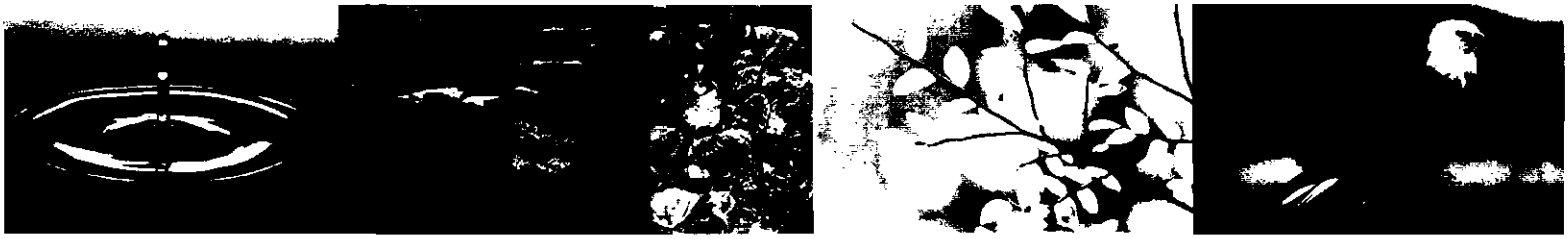
Environmental Protection Mgr

TITLE

DATE

# Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055



# Quality Assurance Project Plan Addendum No. 4

for

# PCB Capacitor Burial/Pole Yard Building 2721 Investigation (Phase 5) SWMU 17

## Naval Surface Warfare Center Crane Crane, Indiana

## Contract Task Order 0020

## August 2008

NAVFAC

Midwest
201 Decatur Avenue
Building IA, Code EV
Great Lakes, Illinois 60088

TETRA TECH NUS, Inc

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# ACRONYMS

| | |
|---|---|
| °C | Degrees Celsius |
| bgs | Below ground surface |
| CLEAN | Comprehensive Long-Term Environmental Action Navy |
| CTO | Contract Task Order |
| DO | Dissolved oxygen |
| FOL | Field Operations Leader |
| HASP | Health and Safety Plan |
| IDW | Investigation-derived waste |
| IM | Interim Measure |
| IMWP | Interim Measures Work Plan |
| mg/kg | Milligrams per kilogram |
| MS | Matrix spike |
| MSD | Matrix spike duplicate |
| NAVFAC SE | Naval Facilities Engineering Command, Southeast |
| NSWC | Naval Surface Warfare Center |
| NTU | Nephelometric Turbidity Unit |
| ORP | Oxidation-reduction potential |
| OSHA | Occupational Safety and Health Administration |
| PCB | Polychlorinated biphenyl |
| PPE | Personal protective equipment |
| QA | Quality assurance |
| QC | Quality control |
| QAPP | Quality Assurance Project Plan |
| RCRA | Resource Conservation and Recovery Act |
| RFI | RCRA Facility Investigation |
| SAP | Sampling and Analysis Plan |
| SOP | Standard Operating Procedure |
| SSO | Site Safety Officer |
| SWMU | Solid Waste Management Unit |
| TOM | Task Order Manager |
| TtNUS | Tetra Tech NUS, Inc. |
| U.S. EPA | United States Environmental Protection Agency |

# 1.0 PROJECT DESCRIPTION

The Naval Surface Warfare Center (NSWC) Crane prepared a Quality Assurance Project Plan (QAPP) [Tetra Tech, NUS, Inc. (TtNUS), 2001] for a Phase III Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at Solid Waste Management (SWMU) 17 to provide data regarding polychlorinated biphenyl (PCB) contamination in surface and subsurface soil at the PCB Capacitor Burial/Pole Yard. Following approval of the QAPP, TtNUS began site activities in March 2002 as described further in Section 1.4 of this document.

In June 2005, the Navy prepared a QAPP Addendum (TtNUS, 2005) to address data gaps regarding PCB contamination in surface soil, subsurface soil, perched groundwater, surface water, and sediment. See Section 1.4 of this document regarding the field investigation related to this QAPP Addendum.

Based on the previous field investigations in which excessive risk was identified related to PCB contamination at SWMU 17, the Navy prepared a QAPP Addendum No. 2 (TtNUS, 2006a) to identify the full extent of the contamination specifically in ditches 1 through 10 leading into Boggs Creek, Boggs Creek headwaters, Boggs Creek plunge pool, and upper portion of Boggs Creek itself.

Due to PCB contamination found within the headwaters of Boggs Creek, the Navy prepared a QAPP Addendum No. 3 (TtNUS, 2006b) to gather data that would allow the RFI report to be completed, including the delineation of the nature and extent of PCB contamination, primarily in the area around Building 2721 and the tributary flowing west from Building 2721 to Boggs Creek.

As mentioned in the preceding paragraphs, several phases of investigation have previously been conducted at SWMU 17 regarding the PCB contamination. It is the purpose of this QAPP Addendum No. 4 to present the additional proposed field sampling activities at SWMU 17 to determine the full extent of PCB contamination in regards to the surface water and sediment associated with Boggs Creek to its termination into Lake Gallimore. The purpose of these additional sample activities is to determine whether PCB contamination exists in the surface water and/or sediment further downstream in Boggs Creek where it exits SWMU 17, south to its termination into Lake Gallimore. The previous phases of investigation conducted at SWMU 17 are discussed in more detail in Section 1.4.

The role of this QAPP Addendum No. 4 is explained in the following section.

## 1.1 DESCRIPTION OF QAPP ADDENDUM NO. 4

This QAPP Addendum No. 4 will govern the sampling and analyses of surface water and sediment within Boggs Creek and its tributaries to its termination at Lake Gallimore. PCB contamination has been documented in certain sections of upper Boggs Creek (TtNUS, 2007), but the extent of the contamination, specifically in the mid to lower region of the creek, has not yet been fully determined. The intent of this QAPP Addendum No. 4 is to provide the procedures for collection of additional surface water and sediment samples within an area of Boggs Creek that has not previously been investigated to determine if PCB contamination exists. The length of the creek covered by this phase will be approximately 10 miles beginning at the previous most southern sample location (17SD069) and ending at the termination of Boggs Creek at Lake Gallimore. The information collected will fill data gaps regarding potential PCB contamination that exist along the central and southern portion of Boggs Creek and SWMU 17 drainage channels. Upon completion of this field sampling event, the information gathered will be incorporated into the SWMU 17 RFI Report as an addendum. If PCBs are present in concentrations that warrant remediation, they will be addressed in the SWMU 17 Interim Measures Work Plan (IMWP).

The specific objectives to be achieved during this phase of field sampling are as follows:

- Based on additional sampling of surface water and sediment in Boggs Creek, determine if any PCB contamination located downstream of the headwaters of Boggs Creek exceed the 1 ppm decision criteria agreed to by the U.S. EPA, U.S. Fish and Wildlife Service (FWS), and the Navy.

- Provide information necessary to complete the human health and ecological risk assessments for SWMU 17. Laboratory analytical data meeting the human health and ecological screening levels is necessary. Table 1-1 provides the method detection limits and risk criteria for PCB aroclors in both aqueous and sediment matrices.

This QAPP Addendum No. 4 was prepared for the NSWC Crane facility located in Crane, Indiana for the Naval Facilities Engineering Command (NAFVAC) Midwest under Contract Task Order (CTO) 0020 of the Comprehensive Long-Term Environmental Action Navy (CLEAN) IV Contract Number N62467-04-D-0055. This QAPP Addendum No. 4 generally does not repeat information already provided in the approved SWMU 17 QAPP (TtNUS, 2001). Figure 1-1 shows the general location of NSWC Crane and SWMU 17, and Figure 1-2 shows the location of Boggs Creek originating near Building 2721 at SWMU 17 and terminating at Lake Gallimore along the south-central boundary of NSWC Crane.

This QAPP Addendum No. 4 consists of three sections: Section 1.0 is this introduction, which includes a brief description of the proposed sample locations and summarizes previous investigations. Section 2.0 includes the project organization and responsibilities. Section 3.0 includes the Sampling and Analysis Plan (SAP).

## 1.2 NSWC CRANE SITE DESCRIPTION

NSWC Crane is located in the southern portion of Indiana, approximately 75 miles southwest of Indianapolis and 71 miles northwest of Louisville, Kentucky, immediately east of Crane Village and Burns City (Figure 1-1). NSWC Crane encompasses 62,463 acres (approximately 98 square miles), most of which are located in the northern portion of Martin County. Smaller portions of NSWC Crane are located in Greene, Daviess, and Lawrence Counties. NSWC Crane is located in a rural, sparsely populated area. Most of NSWC Crane is forested, and the surrounding area is wooded or farmed land.

NSWC Crane provides material, technical, and logistical support to the Navy for equipment, shipboard weapons systems, and nonexpendable ordnance items. In addition, NSWC Crane supports the Crane Army Ammunition Activity with production, renovation, storage, shipment, demilitarization, and disposal of conventional ammunition.

## 1.3 SWMU 17 SITE DESCRIPTION

SWMU 17 is located in the north-central portion of NSWC Crane as shown on Figure 1-1. The PCB Capacitor Burial/Pole Yard/Building 2721 has been in use since before 1966. Historically, SWMU 17 has been used for the following:

- Storage of electrical capacitors, some of which contained PCBs.
- Storage of electrical transformers, some of which contained PCBs.
- Burial of capacitors, some of which may have contained PCBs.
- Storage of creosote-impregnated utility poles, some of which may have been contaminated with PCBs as a result of leaking transformers.

It has been reported that capacitors were buried at SWMU 17 in the early to mid-1970s, but it is not known whether any capacitors were buried before the early 1970s or after the mid-1970s. The soil at SWMU 17 has been investigated extensively. A geophysical survey was completed as part of the RFI in March of 2002. The survey, conducted to a maximum depth of 10 feet, identified several anomalies as well as a waste burial area and many locations of subsurface metal, including buried utilities.

Subsequently, an Interim Measure was conducted in September 2004. During the Interim Measures, PCB contaminated soil was removed for disposal and excavation for buried capacitors was completed to a depth of between 7 to 12 feet. A large number of electrical insulators and other debris were uncovered, but no capacitor pieces were found in any of the 10 holes that were excavated.

Following is a listing of the SWMU 17 investigations and remedial activities performed to date:

- SAIC soil investigation, March 2001
- Tetra Tech NUS, Inc. (TtNUS) RFI, March 2002
- TolTest IM, September 2004
- TtNUS RFI Addendum field investigation, October 2005
- TtNUS RFI Addendum field investigation, April 2006
- TtNUS RFI Addendum field investigation, May 2006
- TtNUS RFI Addendum field investigation, October 2006

These environmental investigations and the previous IM conducted at SWMU 17 are described in more detail in the following section.

## 1.4 PREVIOUS INVESTIGATIONS

Previous soil investigations at the PCB Capacitor Burial/Pole Yard are summarized below.

**2001 Soil Investigation** – In March 2001, SAIC conducted a soil investigation in and around the SWMU 17 capacitor burial and pole storage areas. The investigation included the collection of surface soil samples only from 0 to 6 inches and 6 to 12 inches below ground surface (bgs). Six composite samples consisting of soil taken from 8 grab locations were collected and analyzed for PCBs only. The results indicated elevated concentrations of PCBs within the surface soils of the investigated areas. The primary contaminant of concern was determined to be Aroclor-1260.

**2002 RFI** – Based on the results of the 2001 soil investigation, TtNUS conducted an RFI for SWMU 17. Field work for the RFI began in March 2002 and consisted of collecting surface and subsurface soil samples and performing a geophysical survey. In total, 44 surface soil samples were collected from 0- to 24 inches bgs, and 44 subsurface soil samples were collected 2 to 4 feet bgs. The sampling identified five areas of concern immediately north and west of Building 357 and within the northwestern drainageway. In addition to soil sampling, a geophysical survey was conducted to identify the limits of the

reported capacitor burial area. The geophysical survey identified several anomalies in the reported capacitor burial area and many locations of subsurface metal including buried utilities.

**2004 Interim Measure** – Based on the results of the 2002 RFI, TolTest was contracted to remove PCB-contaminated soil from the SWMU 17 area. This IM was conducted in September 2004 and resulted in the excavation of 2,140 tons of soil with PCB concentrations less than 25 milligrams per kilogram (mg/kg) and 790 tons of soil with PCB concentrations greater than 25 mg/kg. The excavation around the capacitor burial area uncovered electrical insulators and miscellaneous debris. A small step-down transformer was also found during a search of an adjacent ditch. The transformer was sealed and therefore not sampled for PCBs. It was placed in a drum along with the other sealed capacitors and sent off for proper disposal as part of Crane's routine PCB management program. Because of contractual limitations, excavation operations ceased before all contaminated soil was removed. Site delineation samples and post-excavation sampling indicated that PCBs remained in subsurface soil below 2 feet of clean fill at concentrations greater than 25 mg/kg, and in surface soil within the drainageway downgradient of the disposal area and in the area between Building 357 and the asphalt paved road (eastern end of building) at concentrations greater than 1 mg/kg. However, the sampling did not identify the extent of the remaining contamination. The surface soil within the drainageway downgradient of the disposal area was not excavated because the area was beyond the identified work area and posed no health risk to workers. The surface soil between Building 357 and the paved road at the eastern end of Building 357 was not excavated based on a regulatory decision (TolTest, 2004). It was identified in the TolTest report that these areas would be targeted for further evaluation in a future RFI.

**RFI Addendum Sampling** – In October 2005, April 2006, and May 2006, TtNUS conducted sediment sampling within the drainage channels that receive runoff from the SWMU 17 area and surface soil sampling around Building 357. The October 2005 sampling event included the collection of seven sediment samples from within the Northwest Drainageway, Boggs Creek, Ditch 8, and Ditch 3 and surface and subsurface soil samples around Building 357 (see Figure 1-3). The results identified PCB concentrations ranging from non-detect to 37 mg/kg. The April 2006 sampling event included the collection of 33 sediment samples from within the upstream drainageways originating at SWMU 17. The results identified PCB concentrations ranging from non-detect to 4.6 mg/kg. PCBs were detected at 1.3 mg/kg at location 17SD14, upstream of a contributing stream that originated within SWMU 17. This detection suggested an additional source of PCB contamination other than SWMU 17 in the tributary. Further investigation of facilities upstream of sample location 17SD14 indicated that Building 2721 was historically used as a transformer maintenance facility and that PCB-contaminated oils were discharged through an oil-water separator to the drainage ditch. Consequently, in May 2006, 23 additional sediment samples were collected in the drainage ditch, the stream in which 17SD14 was located, and other

drainage channels upgradient of SWMU 17. The analytical results identified PCB concentrations ranging from non-detect to 3.3 mg/kg.

As a result of finding additional PCB contamination in the stream originating at Building 2721, additional soil and sediment samples were collected in the vicinity of Building 2721 in October 2006, within areas along the stream outside its banks that would likely receive flood waters, and within the drainage ditches in the vicinity of SWMU 17 not previously sampled. In total, 62 additional samples were collected from 35 soil boring and five sediment locations. PCB concentrations in the soil samples collected around Building 2721 and in the potential flood zone along the stream north of SWMU 17 ranged from non-detect to 73 mg/kg. The maximum PCB concentration was detected in a surface soil sample (0 to 2 feet bgs) collected from soil boring 17SB55, which is located adjacent to the southwestern corner of Building 2721. PCB concentrations in sediment samples collected from the streams and drainage channels ranged from non-detect to a high of 74.16 mg/kg detected in sample 17SD61. Figures 1-4 and 1-5 present the most recent soil and sediment sampling locations and their associated PCB concentrations.

## 1.5 EXTENT OF CONTAMINATION

The 2004 IM removed the majority of PCB (Aroclor-1260) contamination from the SWMU 17 capacitor burial area and pole storage yard. However, verification samples collected following the IM identified isolated areas where PCB contamination remained in subsurface soil. In addition, as indicated by the 2005 and 2006 sediment sampling events, PCB contamination is present in sediment and soil within the drainage channel network surrounding SWMU 17 and surface and subsurface soil surrounding Buildings 357 and 2721.

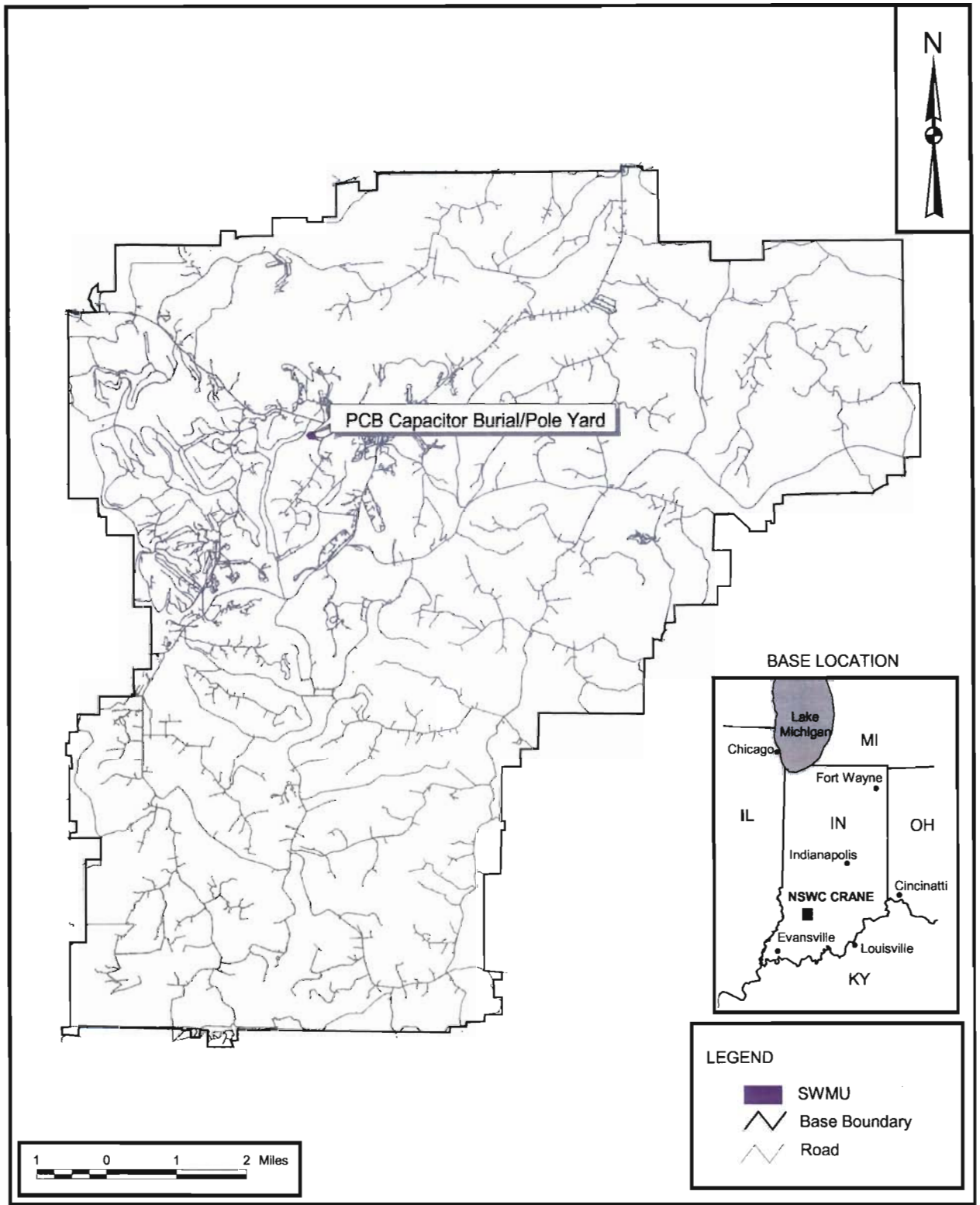
TABLE 1-1

METHOD DETECTION LIMITS AND RISK CRITERIA
QAPP ADDENDUM NO. 4
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
NSWC CRANE
CRANE, INDIANA

| PARAMETER | CAS NUMBER | Aqueous Matrix | | | Solid Matrix | | | |
|---|---|---|---|---|---|---|---|---|
| | | Laboratory | | Risk-Based Target Level [2] (µg/L) | Laboratory | | Risk-Based Soil Target Level [2] (mg/kg) | Risk-Based Sediment Target Level [2] (mg/kg) |
| | | TV [1] (µg/L) | RL [1] (µg/L) | | TV [1] (mg/kg) | RL [1] (mg/kg) | | |
| **POLYCHLORINATED BIPHENYLS (SW-846 METHOD 8081B/8082A)** | | | | | | | | |
| Aroclor-1016 | 12674-11-2 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |
| Aroclor-1221 | 11104-28-2 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |
| Aroclor-1232 | 11141-16-5 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |
| Aroclor-1242 | 53469-21-9 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |
| Aroclor-1248 | 12672-29-6 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |
| Aroclor-1254 | 11097-69-1 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |
| Aroclor-1260 | 11096-82-5 | 0.25 | 0.5 | 0.00012 | 0.25 | 0.0166 | 0.000332 | 0.0598 |

µg/L = Micrograms per liter.
mg/kg = Milligrams per kilogram.
RL = Reporting Limit.
TV = Threshold value (best estimate of realistic MDL basd on statsitical calculations and laboratory experience).

1 Threshold values and reporting limits may change prior to commencement of sampling activities.
2 Value is based on the lowest human health or ecological risk-based criterion as presented in Appendix B, Table B-1 (aqueous) and B-2 (solids) (TtNUS, 2004).
3 Shading indicates those chemicals for which the threshold value and/or reporting limit exceeds the risk-based target level for the project.

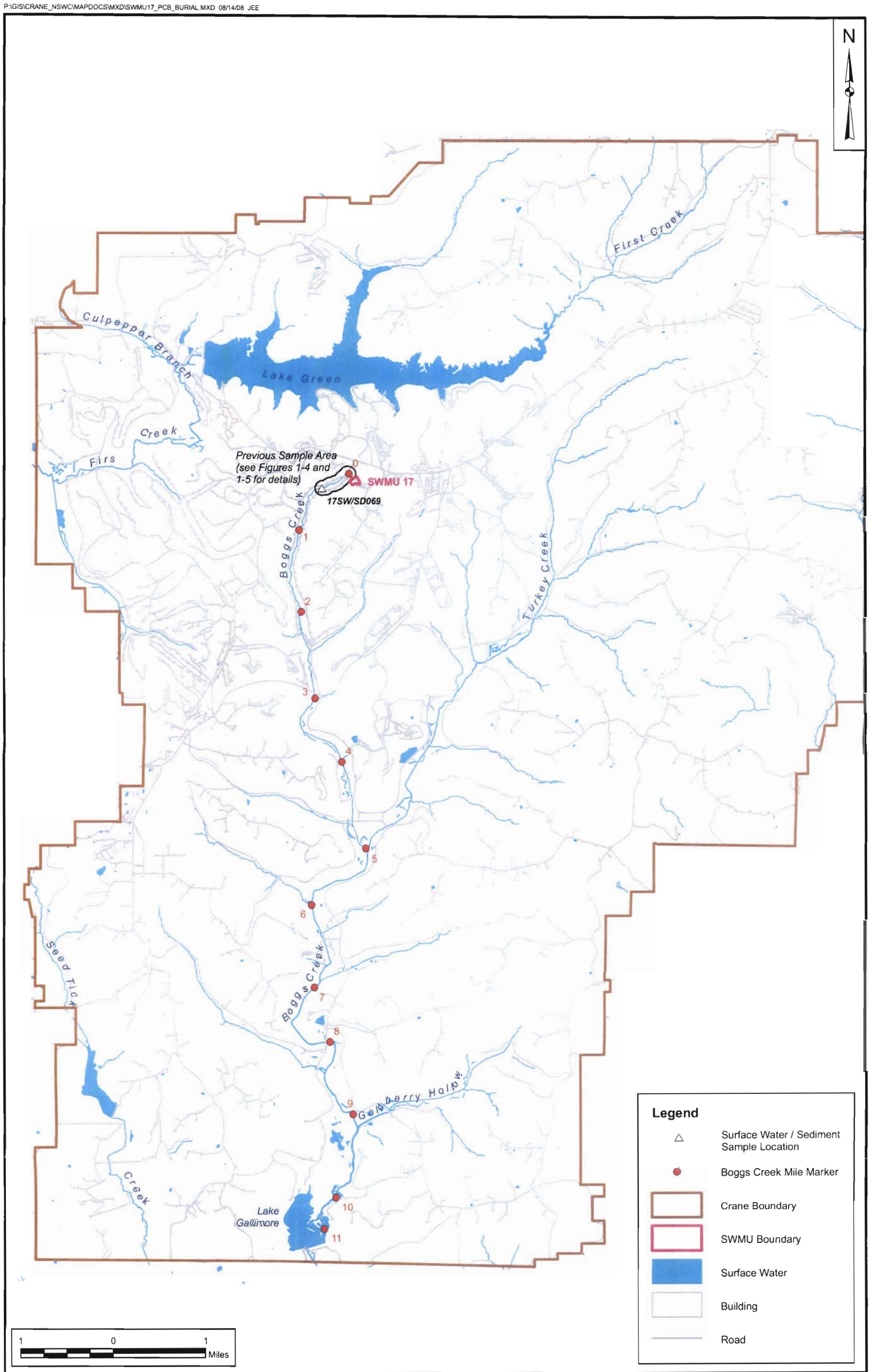



| DRAWN BY | DATE | | | CONTRACT NUMBER |
|---|---|---|---|---|
| J. LAMEY | 5/6/02 | | | 4070 |
| CHECKED BY | DATE | | | APPROVED BY / DATE |
| C. RUMER | 08/12/08 | | | J. GOERDT / 12/27/07 |
| COST/SCHEDULE-AREA | | | | APPROVED BY / DATE |
| | | | | — / — |
| SCALE | | | | DRAWING NO. / REV |
| AS NOTED | | | | FIGURE 1-1 / 0 |

SITE LOCATION MAP
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
NAVAL SURFACE WARFARE CENTER
CRANE, INDIANA

P:\GIS\NSWC_CRANE\APR\POLE_YARD.APR SITE LOCATION 12/27/07 JEE

P:\GIS\CRANE_NSWC\MAPDOCS\MXD\SWMU17_PCB_BURIAL.MXD 08/14/08 JEE




| DRAWN BY | DATE | CONTRACT NUMBER |
|---|---|---|
| J. ENGLISH | 08/14/08 | CTO 0376 |
| CHECKED BY | DATE | APPROVED BY / DATE |
| C. RUMER | 08/14/08 | — / — |
| REVISED BY | DATE | APPROVED BY / DATE |
| — | — | — / — |
| SCALE | | FIGURE NO. / REV |
| AS NOTED | | FIGURE 1 - 2 / 0 |

NAVFAC

SWMU 17 - PCB CAPACITOR BURIAL / POLE YARD

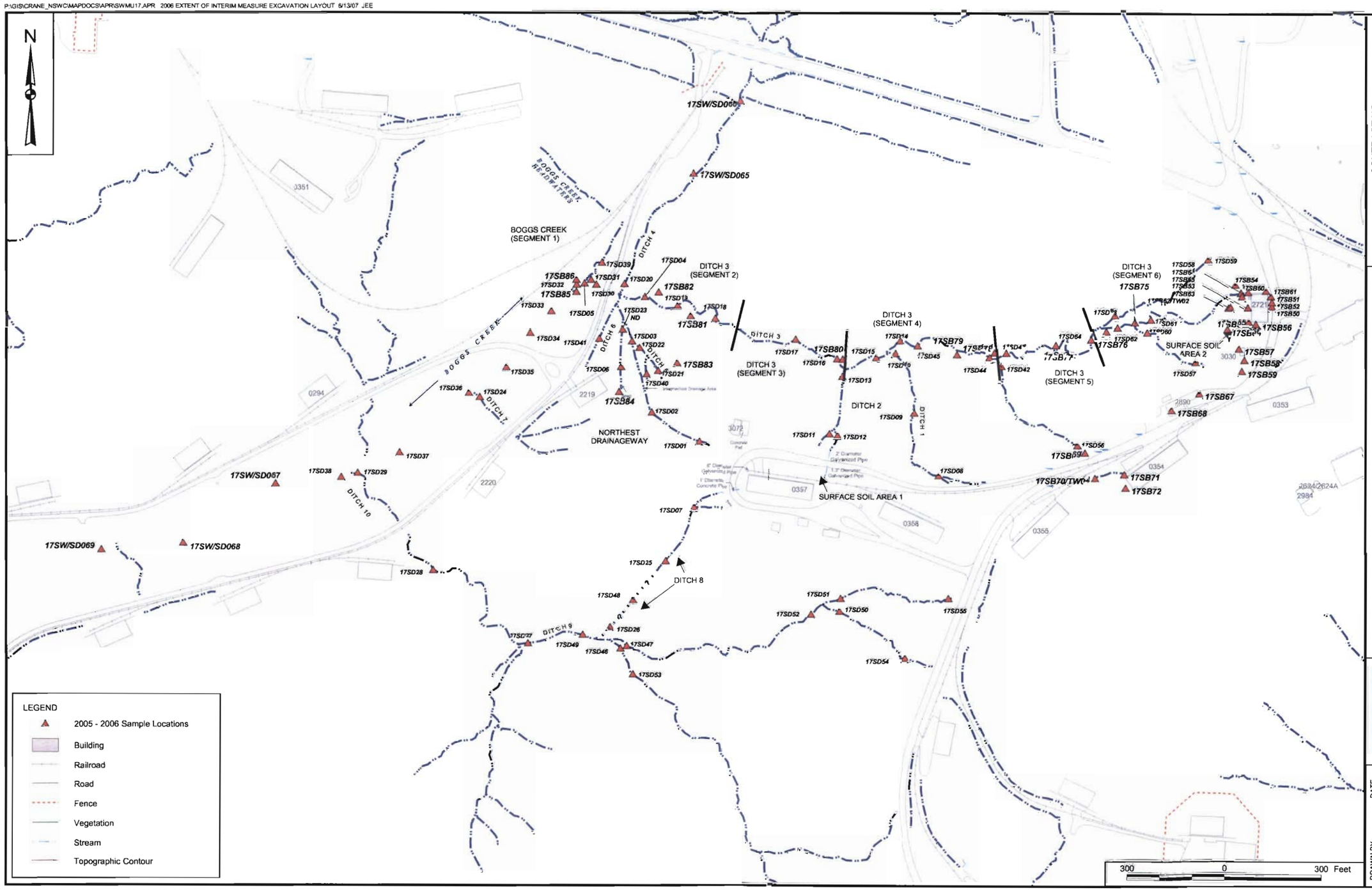
QAPP ADDENDUM NO. 4

BOGGS CREEK

NSWC CRANE

CRANE, INDIANA

P:\GIS\CRANE_NSWC\MAPDOCS\APR\SWMU17.APR 2006 EXTENT OF INTERIM MEASURE EXCAVATION LAYOUT 6/13/07 JEE




CONTRACT NO. 0020

APPROVED BY ____ DATE ____

APPROVED BY ____ DATE ____

DRAWING NO. FIGURE 1 - 3

REV 0

2005 AND 2006 SAMPLE LOCATIONS
SWMU 17 - PCB CAPACITOR BURIAL / POLE YARD
QAPP ADDENDUM NO. 4
NSWC CRANE
CRANE, INDIANA

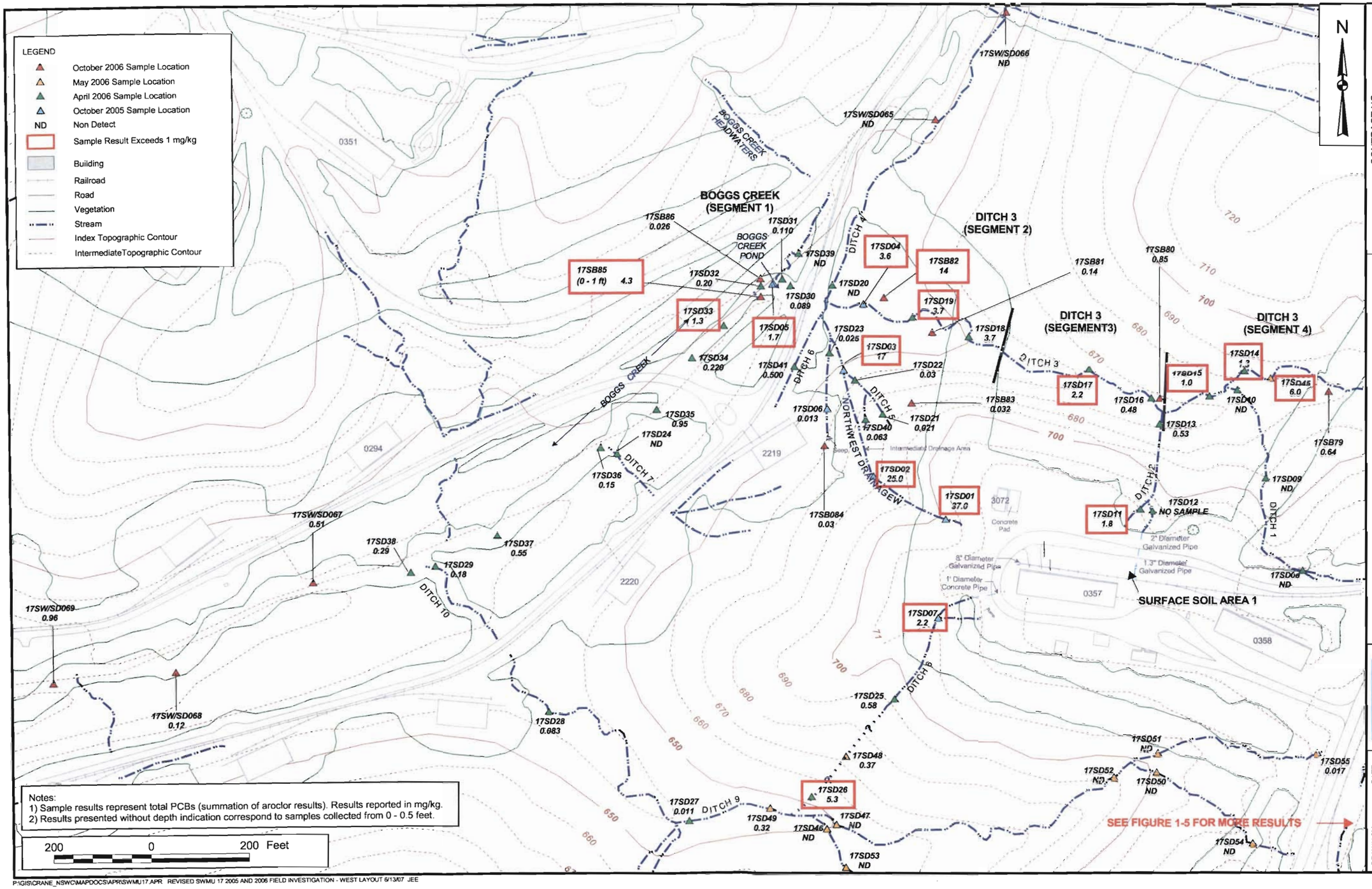
NAVFAC

DRAWN BY S. PAXTON DATE 2/23/07

CHECKED BY T. RILEY DATE 6/13/07

COST/SCHED-AREA

SCALE AS NOTED




P:\GIS\CRANE_NSWC\MAPDOCS\APR\SWMU17.APR REVISED SWMU 17 2005 AND 2006 FIELD INVESTIGATION - WEST LAYOUT 6/13/07 JEE

CONTRACT NO. 0020

APPROVED BY ___ DATE ___

APPROVED BY ___ DATE ___

DRAWING NO. FIGURE 1 - 4

REV 0

TtNUS 2005 AND 2006 FIELD INVESTIGATIONS
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC, CRANE
CRANE, INDIANA

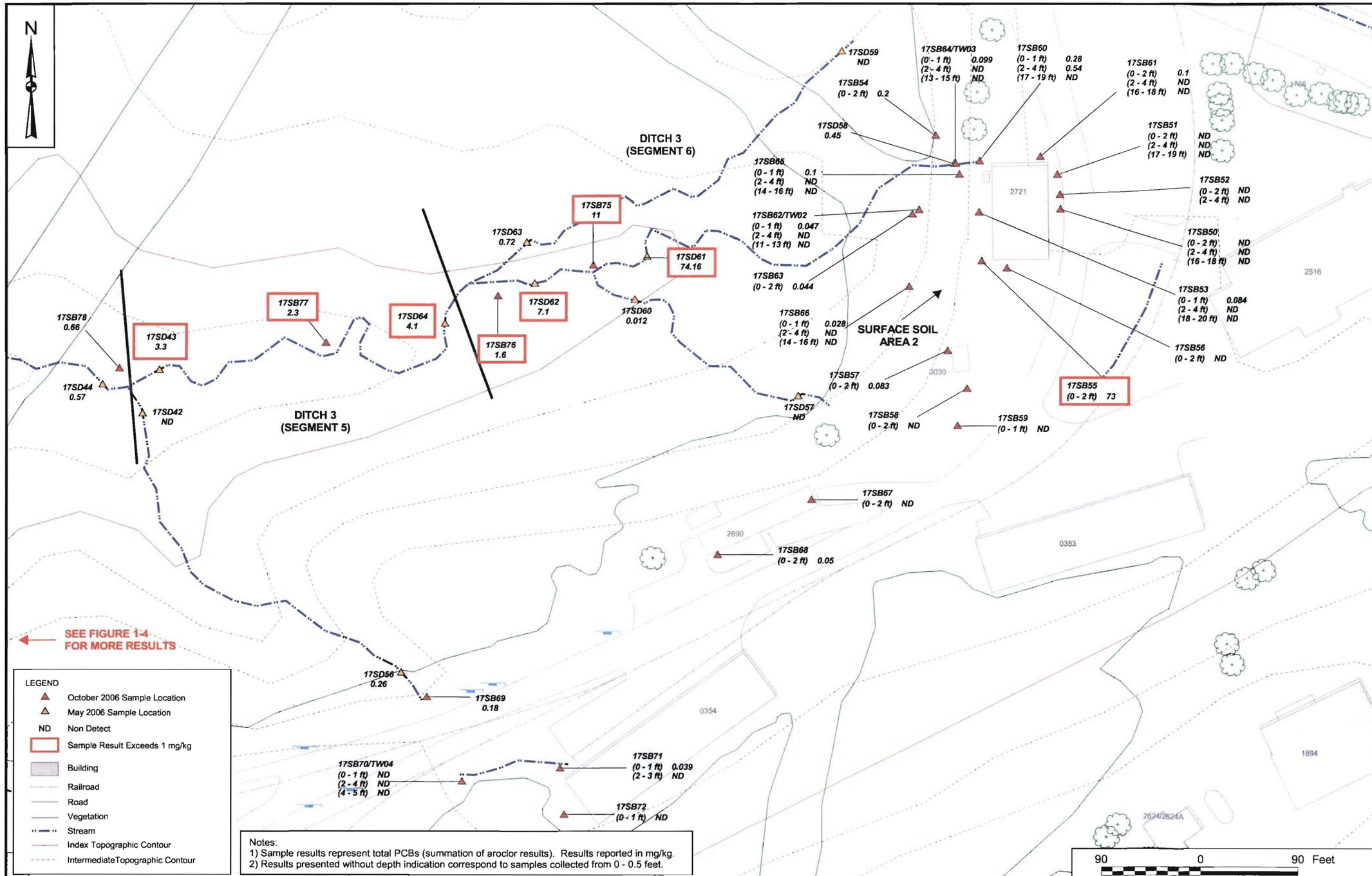
NAVFAC

DRAWN BY S. PAXTON DATE 2/23/07

CHECKED BY J. GOERDT DATE 6/13/07

COST/SCHED-AREA

SCALE AS NOTED




Notes:
1) Sample results represent total PCBs (summation of aroclor results). Results reported in mg/kg.
2) Results presented without depth indication correspond to samples collected from 0 - 0.5 feet.

P:\GIS\CRANE_NSWC\MAPDOCS\APR\SWMU17.APR REVISED SWMU 17 2005 AND 2006 FIELD INVESTIGATION EAST LAYOUT 6/13/07 JEE

CONTRACT NO. 0020

APPROVED BY ___ DATE ___

APPROVED BY ___ DATE ___

DRAWING NO. FIGURE 1 - 5

REV 0

TiNUS 2005 AND 2006 FIELD INVESTIGATIONS
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC, CRANE
CRANE, INDIANA

NAVFAC

DRAWN BY S. PAXTON DATE 2/23/07

CHECKED BY J. GOERDT DATE 6/13/07

COST/SCHED-AREA

SCALE AS NOTED

# 2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section presents the project management and organization for conducting field operations at NSWC Crane SWMU 17. Staffing and coordination requirements are described in Section 2.0 of the QAPP Addendum No. 1 (TtNUS, 2006). The authorities and organizational relationships of key personnel are depicted on Figure 2-1. Corresponding addresses and telephone numbers of key personnel are listed by organization in Table 2-1.

Pace Analytical Services, Inc. of Seattle, Washington, will perform all sample analyses for this field activity.

The subcontracted laboratory is responsible for analyzing all samples in accordance with the analytical methods and additional requirements specified in this QAPP Addendum. It will also be the responsibility of the analytical laboratory to properly dispose of unused sample aliquots. Responsibilities of key laboratory personnel are outlined in Section 2.2.4 of the QAPP Addendum No. 1 (TtNUS, 2006).

TABLE 2-1

KEY PROJECT PERSONNEL NAMES,
PHONE NUMBERS, AND ADDRESSES
QAPP ADDENDUM NO. 4
SWMU 17 – PCB CAPACITOR BURIAL/POLE YARD
NSWC CRANE
CRANE, INDIANA


| PERSON / TITLE / ORGANIZATION | ADDRESS | TELEPHONE |
|---|---|---|
| Doug Griffin<br>Corrective Action Project Manager<br>Office of Land Quality<br>Hazardous Waste Permits<br>IDEM | Corrective Action Section<br>Office of Land Quality<br>Hazardous Waste Permits<br>100 North Senate Avenue<br>P.O. Box 6015<br>Indianapolis, Indiana 46206-6015 | Phone: (313) 233-2710 |
| Craig Pender<br>Project Chemist<br>Office of Land Quality<br>IDEM | Office of Land Quality<br>Hazardous Waste Permits<br>100 North Senate Avenue<br>P.O. Box 6015<br>Indianapolis, Indiana 46206-6015 | Phone: (313) 234-0941 |
| Peter Ramanauskas<br>U.S. EPA Oversight<br>U.S. EPA Region 5 | U.S. EPA Region 5<br>77 West Jackson Blvd.<br>Chicago, Illinois 60604 | Phone: (312) 886-7890<br>FAX: (312) 353-4788 |
| Howard Hickey<br>Remedial Project Manager<br>U.S. Navy<br>NAVFAC | Department of the Navy<br>Building 1A, Code 45313<br>201 Decatur Avenue<br>Great Lakes, IL 60080 | Phone: (847) 688-5999<br>FAX: (847) 688-2319 |
| Tom Brent<br>Environmental Manager<br>NSWC Crane | NSWC Crane<br>Code PRCR4-TB<br>B-3260<br>300 Highway 361<br>Crane, Indiana 47522-5009 | Phone: (812) 854-6160<br>FAX: (812) 854-4177 |
| John Trepanowski<br>Program Manager<br>Tetra Tech NUS | Tetra Tech NUS<br>234 Mall Boulevard, Suite 260<br>King of Prussia, Pennsylvania 19406 | Phone: (610) 491-1532<br>FAX: (610) 491-9688 |
| Matt Soltis<br>Health and Safety Manager<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-8912<br>FAX: (412) 921-4040 |
| Steve Ruffing<br>Project Manager<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-8989<br>FAX: (412) 921-4040 |
| James Goerdt<br>Field Operations Leader<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-8425<br>FAX: (412) 921-4040 |
| Joseph Samchuck<br>Data Validation Manager<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-8510<br>FAX: (412) 921-4040 |

FIGURE 2-1

PROJECT ORGANIZATION CHART
SWMU 17 – PCB CAPACITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC CRANE
CRANE, INDIANA

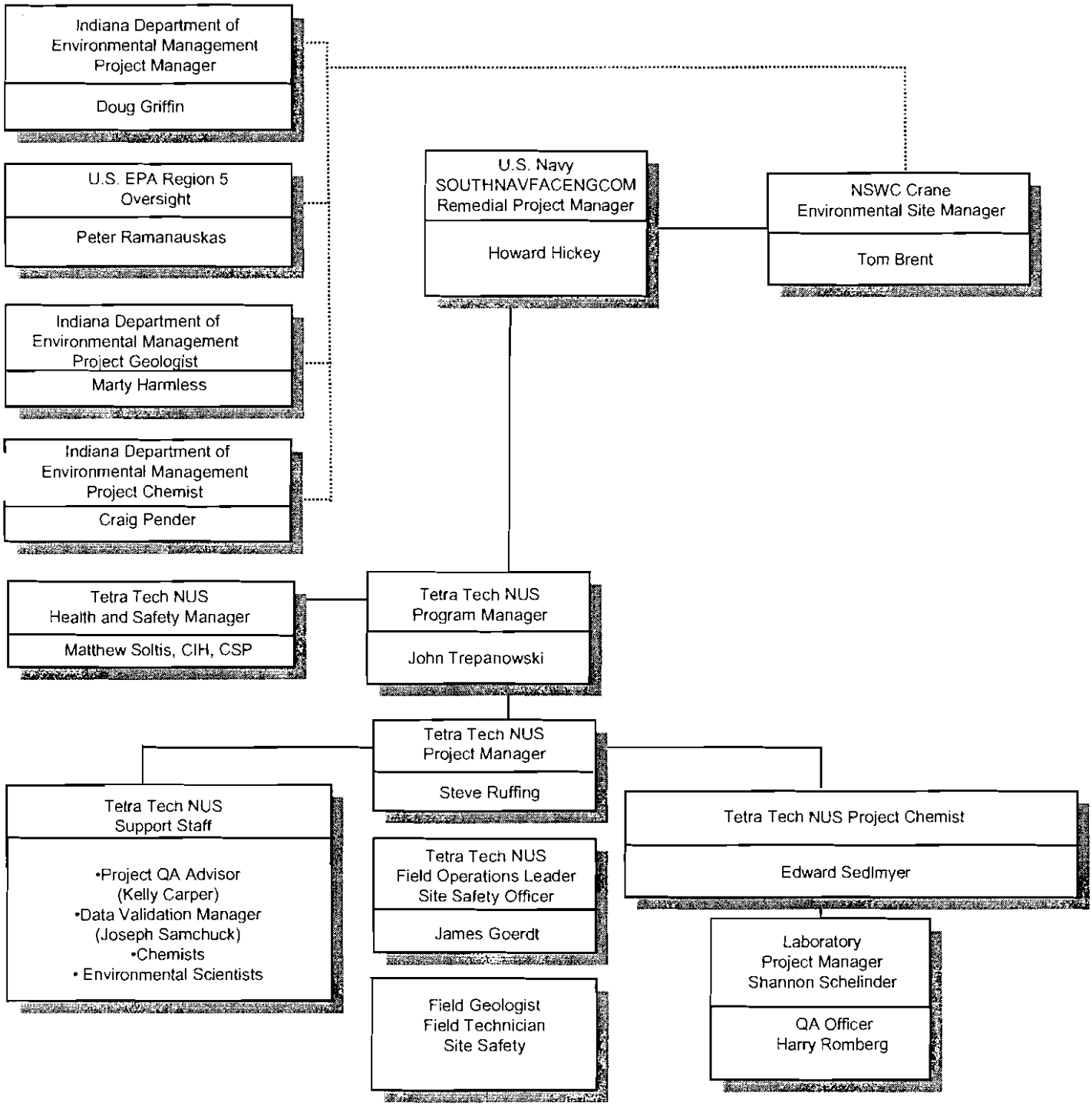


TABLE 2-1

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SWMU 17 – PCB CAPACITOR BURIAL/POLE YARD
NSWC CRANE
CRANE, INDIANA


| PERSON / TITLE / ORGANIZATION | ADDRESS | TELEPHONE |
|---|---|---|
| Edward Sedlmyer<br>Project Chemist<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-8704<br>FAX: (412) 921-4040 |
| Kelly Carper<br>Quality Assurance Advisor<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-7273<br>FAX: (412) 921-4040 |
| Shannon Schelinder<br>Project Manager<br>PACE Analytical Services, Inc. | PACE Analytical Services, Inc.<br>940 South Harney Street<br>Seattle, Washington 98108 | Phone: (206) 957-2449<br>FAX: (206) 767-5063 |
| Harry Romberg<br>Laboratory Quality Assurance Manager<br>PACE Analytical Services, Inc. | PACE Analytical Services, Inc.<br>940 South Harney Street<br>Seattle, Washington 98108 | Phone: (206) 767-5060<br>FAX: (206) 767-5063 |

TABLE 2-1

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|---|---|---|
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| Peter Ramanauskas<br>U.S. EPA Oversight<br>U.S. EPA Region 5 | U.S. EPA Region 5<br>77 West Jackson Blvd.<br>Chicago, Illinois 60604 | Phone: (312) 886-7890<br>FAX: (312) 353-4788 |
| Howard Hickey<br>Remedial Project Manager<br>U.S. Navy<br>NAVFAC | Department of the Navy<br>Building 1A, Code 45313<br>201 Decatur Avenue<br>Great Lakes, IL 60080 | Phone: (847) 688-5999<br>FAX: (847) 688-2319 |
| Tom Brent<br>Environmental Manager<br>NSWC Crane | NSWC Crane<br>Code PRCR4-TB<br>B-3260<br>300 Highway 361<br>Crane, Indiana 47522-5009 | Phone: (812) 854-6160<br>FAX: (812) 854-4177 |
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| Matt Soltis<br>Health and Safety Manager<br>Tetra Tech NUS | Tetra Tech NUS<br>661 Andersen Drive<br>Pittsburgh, Pennsylvania 15220-2745 | Phone: (412) 921-8912<br>FAX: (412) 921-4040 |
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# 3.0 SAMPLING AND ANALYSIS PLAN

## 3.1 GENERAL APPROACH

This section describes locations and rationales, as well as the equipment and procedures to be used for collecting, handling, preserving, and shipping samples to the analytical laboratory. All surface water and sediment samples will be analyzed for PCBs by EPA Method SW 846-8081B/8082A as listed in Tables 3-1 and 3-2, respectively. The text references field Standard Operating Procedures (SOPs) (located in Appendix A) and the updated Health and Safety Plan (HASP) included in Appendix B, where applicable.

The field activities described in this QAPP Addendum No. 4 includes the following:

- Collection of 40 sediment samples along a 10-mile stretch of Boggs Creek. All sediment samples will be composite samples as discussed further in Section 3.3.1.2.
- Collection of six surface water samples along a 10-mile stretch of Boggs Creek.
- Mark all sample locations with global positioning system points.

Prior to any field activities, the Task Order Manager (TOM) will ensure that all field personnel read and understand this QAPP Addendum No. 4 and the associated HASP, the Field Operations Leader (FOL) will ensure that all required field equipment for non-health and safety-related equipment is available and operational, and the Site Safety Officer (SSO) will ensure that all health and safety-related equipment is available and operational.

## 3.2 SAMPLE IDENTIFICATION SYSTEM

All samples collected for fixed-base laboratory analyses will be properly labeled with an adhesive-backed sample label affixed to each sample container in accordance with SOP_CTO0020-01 (Appendix A). The sample labels will include the following information: project name, project location, sample tracking number, sampling date, sampling time, type of analysis required, matrix type, preservative, initials of sampler, and the name of the analytical laboratory to which the sample will be submitted.

Each sample collected will be assigned a unique sample tracking number. The sample tracking number for sediment samples will be a four-segment, alphanumeric code beginning with the site designation ("17" represents SWMU 17) and followed by codes for the sample medium, sample location, and sample depth. The sample tracking number for surface water samples will also be a four-segment, alphanumeric

code identifying the site ("17"), sample medium, sample location, and sampling round. These numbering schemes are described in SOP_CTO0020-02. Previously assigned sample numbers will not be duplicated. Any other pertinent information regarding sample identification will be recorded in the field logbooks and on sample log sheets.

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

Matrix spike (MS) and matrix spike duplicate (MSD) samples will be designated on the field documentation and chain-of-custody forms (see SOP_CTO0020-03).

## 3.3 SAMPLING LOCATIONS, ANALYSES, AND RATIONALES

This section identifies sampling locations, quality assurance (QA)/quality control (QC) samples to be collected, analyses to be performed, and rationales for the sampling and analytical program. Details regarding the preservation, packaging, and shipping of samples are included in Section 3.5. All samples collected will be prepared and analyzed according to the normal laboratory protocol.

The total number of surface water and sediment analyses for each analyte group is tabulated in Tables 3-1 and 3-2, respectively. Surface water and sediment QA/QC samples will be collected at frequencies listed in Table 3-3. Table 3-4 presents a summary of the sample analyses, container types and volumes, preservation requirements, and holding times for the samples to be collected at SWMU 17.

### 3.3.1 Sampling Strategy

Currently, the furthest downstream sample location (17SW/SD069) in Boggs Creek is approximately 1/2 mile downstream from the headwaters originating near Building 2721 (see Figure 1-1). During this field activity the surface water and sediment samples will be collected from an area of approximately 10 linear miles of Boggs Creek between this sample location and Lake Gallimore. The surface water samples will be spaced out evenly along this stretch of Boggs Creek to determine whether PCB contamination is present in total and dissolved phases. The sediment samples will be collected at an average rate of three to five composite samples per linear mile in areas of the creek where sediment traps are found.

Surface water samples will be collected by direct filling of the sample container at sample locations having a turbidity reading of 10 Nephelometric Turbidity Units (NTUs) or less, and surface water samples will be filtered through a 0.45-micron filter at sample locations having a turbidity reading greater than 10 NTUs. All sediment samples will be collected using a disposable plastic trowel or sample location-specific stainless-steel trowel. Details regarding sampling equipment and procedures are included in Section 3.3.1.1 (surface water) and Section 3.31.2 (sediment), and SOPs_CTO0020-05-(sediment) and CTO0020-08 (surface water).

All sample locations will be marked with a tall wooden lath driven into the soil on the creek bank. The stake will have brightly colored flagging attached to it to increase visibility and will be labeled using a waterproof marker with the unique sample location ID. Global positioning system points, in lieu of surveying, will be taken at each individual sample location which will allow for future repeatable investigations or guide in any remedial action. All visual observation results will be noted on the sediment sample log.

Medium-specific sample analytical information is presented in Tables 3-1 and 3-2, and appropriate containers to be used for each sample aliquot are listed in Table 3-4. After the samples are containerized, labeled, and bagged (see SOP_CTO0020-01 and SOP_CTO0020-03), they will be placed in a cooler containing ice until they can be packaged and prepared for shipment (Section 3.5.1). Any investigation-derived waste (IDW) will be handled in accordance with Section 3.8.

#### 3.3.1.1 Surface Water

Six surface water samples will be collected along Boggs Creek approximately every 1 to 1-1/2 miles between previous sample location 17SW/SD069 and Lake Gallimore. All samples will be submitted to a fixed-base laboratory for PCB analysis. The surface water samples will be used to evaluate whether PCB contamination is being carried downstream along the length of Boggs Creek to its termination at Lake Gallimore. Sample collection for surface water will begin at the furthest downstream location near Lake Gallimore and proceed to the farthest upstream location.

Field measurements of water quality parameters including pH, specific conductance, oxidation-reduction potential (ORP), temperature, dissolved oxygen (DO), and turbidity will be collected at all surface water sample locations. These field measurements (excluding turbidity) will be made using a YSI 6-Series Environmental Monitoring System or equivalent type of instrument. Turbidity measurements will be made using a LaMotte turbidity meter or equivalent. Calibration and measurements made with the field instruments will be in accordance with SOP_CTO0020-11.

Each calibration of an instrument will be recorded on an equipment calibration log sheet. Water quality measurements along with date, time, instrument operator, and visual and other observations (e.g., weather conditions) will be recorded on sample collection logs as appropriate.

#### 3.3.1.2 Sediment

Up to 40 composite sediment samples will be collected within Boggs Creek between previous sample location 17SW/SD069 and Lake Gallimore. The samples will be submitted to a fixed-base laboratory for PCB analysis. The sediment samples will be used to evaluate whether PCB contamination is present along this portion of Boggs Creek. Final sample locations will be determined in the field during sample collection based on the general criteria listed below:

- Collection of samples within the defined channel of Boggs Creek.
- Three to five sample locations per mile.
- A minimum separation of 1,000 feet between sample locations.
- Samples to be collected within sediment traps (i.e.: bends in creek or areas of decreased flow velocity due to channel widening).

Each sediment sample will be a composite sample comprised of four sample aliquots. Samples will be collected at a depth of 0 to 6 inches, and if sufficient sediment is available, a second sample will be collected at a depth of 6 to 12 inches. Up to 10 sediment sample locations are proposed for the deeper sediment sampling. When sampling from a specific sediment depositional area or trap, the area will be marked into quadrants with an aliquot sample taken from each quadrant and mixed to make up the composite sample for that particular location. It is assumed the depositional areas will range anywhere from a few square feet up to 10 square feet or more. Temporary individual pin flags will be placed in the sediment trap to mark the four aliquot locations and then photographed. Upon completion of sample collection and photographing at all sample locations, the temporary pin flags will be immediately removed from the site. The individual aliquots will be collected via a disposable plastic trowel and mixed in a 2-gallon disposable ZipLoc® bag. Alternatively, sediment samples may be collected using a stainless-steel spoon and mixed in a stainless-steel bowl. The appropriate sample jar(s) used for analyses will then be filled with the mixed composite sample and properly labeled. The stainless-steel bowl and spoon, if employed, will be decontaminated in the field between each composite sample as described in SOP_CTO0020-04. Any used ZipLoc® bags and disposable trowels will be collected in a trash bag and placed into an authorized dumpster for disposal.

As with the collection of surface water samples, sediment sampling will begin at the furthest downstream location near Lake Gallimore and proceed to the farthest upstream location.

## 3.4 QUALITY CONTROL SAMPLES

QC samples will be collected at the frequencies listed in Table 3-3. One duplicate sample per medium will be collected for every 20 samples. The duplicate samples will be placed in the same types of containers and handled in the same manner as the regular samples. The duplicate samples will be given unique QC sample IDs (see SOP_CTO0020-02). Because surface water samples will be directly filled from the water source, no other sampling equipment will be needed to collect these samples. Hence, no rinsate samples associated with surface water samples will be collected. Due to the limited access to sample locations, it is planned that all composite sediment samples will be collected by plastic disposable trowels and mixed in large disposable plastic baggies. If this is the case, no rinsate samples associated with sediment samples will be collected. MS/MSD samples will be collected and analyzed in the fixed-base laboratory at a rate of 1 per every 20 samples per medium. As samples are added to a cooler, the chain-of-custody form should be updated to include each new sample container (per SOP_CTO0020-03).

Field QC sample requirements for field duplicates and possible equipment rinsate blanks required for this project are described below.

Field Duplicates. Field duplicates are obtained during a single act of sampling and are used to assess the overall precision of the sampling and analysis program. Duplicate samples will be collected at a rate of 1 for every 20 samples of each environmental medium. All duplicate samples will be analyzed for the same parameters in the laboratory as their environmental sample counterparts.

Equipment Rinsate Blanks. Equipment rinsate blanks (or rinsate blanks) will be obtained under representative field conditions by running analyte-free water through sample collection equipment after decontamination and placing the water in the appropriate sample containers for analysis. Equipment rinsate blanks will be collected for non-dedicated equipment. For sediment sampling activities, rinsate blanks will be collected by running analyte-free water over the decontaminated non-dedicated sampling equipment before it is placed in sample jars.

Temperature Blanks. Temperature blanks will be added to each cooler that contain samples which must be kept cooled as a preservation requirement.

## 3.5 SAMPLE HANDLING, PACKAGING, AND SHIPPING

### 3.5.1 Sample Preservation

Preservation requirements for each of the parameters of interest are provided in Table 3-4. All samples are to be cooled to 4 degrees Celsius (°C) ± 2°C; no chemical preservatives are necessary. All samples will be promptly chilled with ice to 4°C ± 2°C and packaged in an insulated cooler. Each cooler will include a temperature blank. Ice will be sealed in containers to prevent water leakage. Samples will not be frozen.

### 3.5.2 Sample Labeling

Before samples are packaged, the sample labels will be checked to ensure that all information on the labels is complete and correct (see SOP_CTO0020-01) and that it matches the information on the sample collection log sheets and chain-of-custody form.

### 3.5.3 Sample Packaging

Each sample container shipped to the laboratory will be placed in a ZipLoc® bag to prevent cross-contamination and leakage. If necessary, each ZipLoc® bag will be wrapped in bubble wrap to prevent breakage and cross-contamination. Only shipping containers that meet all applicable state and federal standards for safe shipment will be used. Cube ice will be placed in and around the samples in sufficient quantity to ensure that the samples remain chilled (4°C ± 2°C) during transport to the analytical laboratory.

SOP_CTO0020-06 provides a detailed description of sample handling, packaging, and shipping procedures required for this project. The FOL will be responsible for ensuring the completion of the following forms:

- Sample labels
- Chain-of-custody forms
- Custody seals for coolers
- Shipping labels for coolers
- Express mail air bills

### 3.5.4 Sample Shipping

Shipping containers (i.e., coolers) will be sealed with nylon strapping tape in at least two places, and custody seals will be signed, dated, and affixed in a manner that will allow the receiver to quickly identify any tampering that may have occurred during transport to the laboratory (see SOP_CTO0020-01 and SOP_CTO0020-03).

Shipment will be made by a public courier at the next scheduled pickup following completion of sample collection. Copies of the air bills will be retained by the FOL for tracking purposes, if needed, and for communications with the laboratory. Air bills will be retained for the permanent record file.

### 3.5.5 Sample Custody

Custody of samples must be maintained and documented at all times, in accordance with SOP_CTO0020-03, beginning with the collection of samples in the field. A chain-of-custody form will be maintained for all samples collected during this investigation.

## 3.6 RECORD KEEPING

Standard forms, field notebooks, and a field logbook or electronic data files will be used to record all sample collection activities, field measurements, observations concerning site conditions, and other project-related information. Additional field records include sample log sheets, daily activity records, field logbooks, drilling and well completion log sheets, and field instrument calibration log sheets, among others. More details regarding record keeping are included in SOP_CTO0020-03.

### 3.6.1 Field Logbooks

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

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

### 3.6.2 Equipment Calibration Logs

An equipment calibration log sheet will be used to record each time an instrument is calibrated, recalibrated, or checked against a standard or background. Field instruments will be calibrated in accordance with the manufacturer's procedures.

The procedures and standards to be used for instrument calibration are discussed in SOP_CTO0020-07.

### 3.6.3 Sample Collection Logs

All sample log sheets will be completed in accordance with SOP_CTO0020-03.

### 3.6.4 Chain-of-Custody Forms

A chain-of-custody form will be maintained for all samples collected during this investigation. These forms are a record of the people having custody of the samples from the time the samples are collected to the time they are analyzed and disposed (see SOP_CTO0020-03). The completed chain-of-custody document will be placed in a sealed plastic envelope, and taped to the top inside lid of the shipping container before it is shipped. A copy of each chain-of-custody form will be retained by the FOL.

### 3.6.5 Shipping Forms/Air Bills

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

### 3.6.6 Permanent Record File

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

## 3.7 EQUIPMENT DECONTAMINATION

All reusable equipment used to collect samples will be decontaminated in accordance with SOP_CTO0020-04.

## 3.8 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

It is anticipated that this investigation will generate the following types of potentially contaminated residues or IDW:

- Personal protective equipment (PPE)
- Equipment decontamination fluids
- Plastic disposable trowels
- Plastic ZipLoc® baggies

IDW will be handled as described in SOP_CTO0020-09.

## 3.9 SAFETY

The health and safety activities described in the following sections will ensure that field activities are performed at a high level of safety.

### 3.9.1 Health and Safety Plan

The HASP used for previous field activities at SWMU 17 and updated for this phase of investigation will be followed. The HASP has been prepared to describe specific health and safety requirements, concerns, and information related to these site activities. This document will be read and understood by each person working at the site. Each worker or visitor to the site must sign an acknowledgment that he or she has read and understands the HASP. The HASP has been included as Appendix B within this document.

### 3.9.2 Health and Safety Training

All workers involved in the field investigation shall have successfully completed the Occupational Safety and Health Administration (OSHA)-mandated 40-hour health and safety training and the follow-up annual 8-hour refresher courses when appropriate.

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

### 3.9.3 Medical Approval and Personal Protective Clothing and Equipment

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

### 3.9.4 Safety Meetings

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

## 3.10 ORGANIZATION AND LOGISTICS OF FIELD INVESTIGATIONS

### 3.10.1 Personnel

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

### 3.10.2 Subcontractors

The Project Chemist will direct the activities of the fixed-base laboratory.

### 3.10.3 Mobilization and Demobilization

Following approval of this QAPP Addendum No. 4, TtNUS will begin mobilization activities. All field team members will review this document and the associated HASP prior to mobilizing to the field. In addition, a field team orientation meeting will be held to familiarize personnel with the scope of the field activities. Items to be presented during that meeting include the following:

- Identification of this QAPP Addendum No. 4, associated HASP, and applicable field SOPs (Appendix A).
- Site-specific safety concerns and requirements.
- Project objectives.
- Sampling designs and strategies.
- Site-specific particulars of field operations (e.g., locations of utilities, physical access to sampling locations, communication mechanisms, lines of authority and responsibility, scheduling requirements, sample shipping concerns, etc.).
- Laboratory coordination.
- Site access requirements.
- Travel requirements.

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

## 3.11 SCHEDULE

Approximately 10 working days have been scheduled to perform the described field activities at SWMU 17.

TABLE 3-1

LISTING OF SURFACE WATER SAMPLES AND LABORATORY ANALYSES
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC CRANE
CRANE, INDIANA

| Sample Location[1] | Sample ID[1] | PCBs |
|---|---|---|
| | | SW-846 8081B/8082A |
| 17SW/SD0XX | 17SW0XX | 1 |
| 17SW/SD0XX | 17SW0XX | 1 |
| 17SW/SD0XX | 17SW0XX | 1 |
| 17SW/SD0XX | 17SW0XX | 1 |
| 17SW/SD0XX | 17SW0XX | 1 |
| 17SW/SD0XX | 17SW0XX | 1 |
| **Total Surface Water Samples** | | **6** |

SW = surface water

1 The surface water samples will be collected at six of the sediment sample locations. The specific sample locations will be determined in the field and the surface water sample IDs will correspond with those particular sediment samples.

TABLE 3-2

LISTING OF SEDIMENT SAMPLES AND LABORATORY ANALYSES
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC CRANE
CRANE, INDIANA


| Sample Location | Sample ID[1] | PCBs |
|---|---|---|
| | | SW-846 8081B/8082A |
| 17SW/SD070 | 17SD0700006 | X |
| | 17SD0700612 | TBD[2] |
| 17SW/SD071 | 17SD0710006 | X |
| | 17SD0710612 | TBD[2] |
| 17SW/SD072 | 17SD0720006 | X |
| | 17SD0720612 | TBD[2] |
| 17SW/SD073 | 17SD0730006 | X |
| | 17SD0730612 | TBD[2] |
| 17SW/SD074 | 17SD0740006 | X |
| | 17SD0740612 | TBD[2] |
| 17SW/SD075 | 17SD0750006 | X |
| | 17SD0750612 | TBD[2] |
| 17SW/SD076 | 17SD0760006 | X |
| | 17SD0760612 | TBD[2] |
| 17SW/SD077 | 17SD0770006 | X |
| | 17SD0770612 | TBD[2] |
| 17SW/SD078 | 17SD0780006 | X |
| | 17SD0780612 | TBD[2] |
| 17SW/SD079 | 17SD0790006 | X |
| | 17SD0790612 | TBD[2] |
| 17SW/SD080 | 17SD0800006 | X |
| | 17SD0800612 | TBD[2] |
| 17SW/SD081 | 17SD0810006 | X |
| | 17SD0810612 | TBD[2] |
| 17SW/SD082 | 17SD0820006 | X |
| | 17SD0820612 | TBD[2] |
| 17SW/SD083 | 17SD0830006 | X |
| | 17SD0830612 | TBD[2] |
| 17SW/SD084 | 17SD0840006 | X |
| | 17SD0840612 | TBD[2] |
| 17SW/SD085 | 17SD0850006 | X |
| | 17SD0850612 | TBD[2] |
| 17SW/SD086 | 17SD0860006 | X |
| | 17SD0860612 | TBD[2] |
| 17SW/SD087 | 17SD0870006 | X |
| | 17SD0870612 | TBD[2] |
| 17SW/SD088 | 17SD0880006 | X |
| | 17SD0880612 | TBD[2] |
| 17SW/SD089 | 17SD0890006 | X |
| | 17SD0890612 | TBD[2] |
| 17SW/SD090 | 17SD0900006 | X |
| | 17SD0900612 | TBD[2] |
| 17SW/SD091 | 17SD0910006 | X |
| | 17SD0910612 | TBD[2] |
| 17SW/SD092 | 17SD0920006 | X |
| | 17SD0920612 | TBD[2] |

TABLE 3-2

LISTING OF SEDIMENT SAMPLES AND LABORATORY ANALYSES
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC CRANE
CRANE, INDIANA


| Sample Location | Sample ID[1] | PCBs |
|---|---|---|
| | | SW-846 8081B/8082A |
| 17SW/SD093 | 17SD0930006 | X |
| | 17SD0930612 | TBD[2] |
| 17SW/SD094 | 17SD0940006 | X |
| | 17SD0940612 | TBD[2] |
| 17SW/SD095 | 17SD0950006 | X |
| | 17SD0950612 | TBD[2] |
| 17SW/SD096 | 17SD0960006 | X |
| | 17SD0960612 | TBD[2] |
| 17SW/SD097 | 17SD0970006 | X |
| | 17SD0970612 | TBD[2] |
| 17SW/SD098 | 17SD0980006 | X |
| | 17SD0980612 | TBD[2] |
| 17SW/SD099 | 17SD0990006 | X |
| | 17SD0990612 | TBD[2] |
| 17SW/SD100 | 17SD1000006 | X |
| | 17SD1000612 | TBD[2] |
| 17SW/SD101 | 17SD1010006 | X |
| | 17SD1010612 | TBD[2] |
| 17SW/SD102 | 17SD1020006 | X |
| | 17SD1020612 | TBD[2] |
| 17SW/SD103 | 17SD1030006 | X |
| | 17SD1030612 | TBD[2] |
| 17SW/SD104 | 17SD1040006 | X |
| | 17SD1040612 | TBD[2] |
| 17SW/SD105 | 17SD1050006 | X |
| | 17SD1050612 | TBD[2] |
| 17SW/SD106 | 17SD1060006 | X |
| | 17SD1060612 | TBD[2] |
| 17SW/SD107 | 17SD1070006 | X |
| | 17SD1070612 | TBD[2] |
| 17SW/SD108 | 17SD1080006 | X |
| | 17SD1080612 | TBD[2] |
| 17SW/SD109 | 17SD1090006 | X |
| | 17SD1090612 | TBD[2] |
| **Total Sediment Samples** | | **50** |

SD = sediment
X = proposed sample
TBD = to be determined

1 All sediment samples will be composite samples of 4 individual aliquots taken from the designated sample location.
2 Each sediment sample location will have one sample at 0 to 6 inches, and up to 10 of the sample locations will have a sample at a depth of 6 to 12 inches.

TABLE 3-3

SUMMARY OF SURFACE WATER AND SEDIMENT ANALYSES AND QUALITY CONTROL SAMPLES
SWMU 17 - PCB CAPICITOR BURIAL/POLE YARD
QAPP ADDENDUM NO. 4
NSWC CRANE
CRANE, INDIANA

| Sample Type | PCBs | |
|---|---|---|
| | SW-8081B/8082A (Sediment) | SW-8081B/8082A (Surface Water) |
| Total Samples | 50 | 6 |
| Field Duplicates (1) | 3 | 1 |
| Rinsate Blanks (2) | 1 | 1 |
| Matrix Spikes (3) | 3 | 1 |
| Matrix Spike Duplicates (4) | 3 | 1 |

1) Field duplicates will be collected at a frequency of 1 per every 20 field samples.
2) Rinsate blanks will be collected at a frequency of one per sampling device or instrument. These are estimates and may vary. If all sediment samples are collected via disposable trowels and baggies, then no associated rinsate blank will be collected. However, a rinsate blank will be collected if stainless-steel trowels are utilized for sediment sample collection. A rinsate blank will be collected for surface water samples if the peristaltic pump and tubing are used in the collection of the samples.
3) Matrix spikes are collected for all organic and inorganic parameters at a frequency of 1 per every 20 field samples.
4) Matrix spike duplicates are collected for all organic parameters. Matrix spike duplicates are collected at a frequency of 1 per every 20 field samples.

## TABLE 3-4

## SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR SURFACE WATER AND SEDIMENT SAMPLES SWMU 17 – PCB CAPACITOR BURIAL/POLE YARD QAPP ADDENDUM NO. 4 NSWC CRANE CRANE, INDIANA

| Parameter | Analyte/Methodology | Medium | Sample Container | Container Volume[1] | Preservation | Maximum Holding Time[2] |
|---|---|---|---|---|---|---|
| PCBs | SW-846 8081B/8082A | Surface Water | Amber glass, Teflon-lined cap | (2) 1 Liter | Cool to 4 °C, dark | Extraction within 7 days; analysis within 40 days of extraction. |
| PCBs | SW-846 8081B/8082A | Sediment | Wide-mouth jar, Teflon-lined cap | (1) 4 ounce | Cool to 4 °C, dark | Extraction within 14 days; analysis within 40 days of extraction. |

1 The number in parenthesis indictes the sample container quantity.
2 All holding times are from date of collection.

°C = Degrees Celsius.

# REFERENCES

Tetra Tech NUS, Inc. (TtNUS), 2001. Quality Assurance Project Plan for PCB Capacitor Burial/Pole Yard Solid Waste Management Unit (SWMU) 17 Resource Conservation and Recovery Act Facility Investigation and Verification. December.

TtNUS, 2005. Quality Assurance Project Plan Addendum for PCB Capacitor Burial/Pole Yard Solid Waste Management Unit (SWMU) 17/04. Resource Conservation and Recovery Act Facility Investigation and Verification of Removal. Naval Surface Warfare Center, Crane. Crane, Indiana, June.

TtNUS, 2006a. Quality Assurance Project Plan Addendum 2 for PCB Capacitor Burial/Pole Yard SWMU 17. Naval Surface Warfare Center, Crane. Crane, Indiana, April.

TtNUS, 2006b. Quality Assurance Project Plan Addendum 3 for Building 2721 Investigation (Phase 4) PCB Capacitor Burial/Pole Yard SWMU 17. Naval Surface Warfare Center, Crane. Crane, Indiana, September.

TtNUS, 2007. Interim Measures Work Plan for SWMU 17 - PCB Capacitor Burial/Pole Yard, NSWC Crane, Crane, Indiana, May.

TolTest (Tol Test Inc.), 2004. Interim Measures Report, PCB Capacitor Burial Pole Yard Remediation, NSWC Crane, Crane, Indiana, November.

# APPENDIX A

# FIELD STANDARD OPERATING PROCEDURES

# APPENDIX A

# FIELD SOP TABLE OF CONTENTS


| | |
|---|---|
| CTO0020-01 | Sample Labeling |
| CTO0020-02 | Sample Identification Nomenclature |
| CTO0020-03 | Sample Custody and Documentation of Field Activities |
| CTO0020-04 | Decontamination of Field Sampling Equipment |
| CTO0020-05 | Composite Sample Collection |
| CTO0020-06 | Sample Preservation, Packaging, and Shipping |
| CTO0020-07 | Calibration and Care of Water Quality Meters |
| CTO0020-08 | Surface Water Sampling |
| CTO0020-09 | Management of Investigation-Derived Waste |
| CTO0020-10 | Estimating Flow in Small Streams |
| CTO0020-11 | Measurement of Water Quality Parameters |

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-01

# SAMPLE LABELING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container. The label for each container will contain identical information.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Writing utensil**

**Disposable medical-grade gloves (e.g. latex, nitrile)**

**Sample logsheets**

**Required sample containers**: All sample containers for analysis by fix-based laboratories will be supplied and deemed certified clean by the laboratory.

**Preprinted sample labels**

**Chain-of-custody records**

**Sealable polyethylene bags**

**Heavy-duty cooler**

**Ice**

## 3.0 PROCEDURES

3.1 The following information will be printed on the labels prior to field activities.

- Project number (CTO 0020)
- Project location (NSWC Crane)
- Sample ID
- Preservative
- Analysis to be performed

- Matrix type
- Laboratory name

3.2 Preprinted sample labels will be prepared before the team mobilizes to the field. Check to determine if

- One sample label exists for each sample container that is to be collected for all media during the field activities.
- The information printed on each label is correct.
- Extra blank labels are brought to the site in case additional environmental samples or QA samples are collected that are not anticipated in the QAPP. Additional blank labels should also be brought to the site in case a sample container is broken or some of the preprinted labels are accidentally lost before they are attached to a container.

3.3 Once at the field site, sample containers should have labels affixed before sampling activities begin.

3.4 Select the labeled containers that are appropriate for a given sample and fill in the date, time, and sampler's initials just before sampling begins. Use a black waterproof marker or pen.

3.5 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.

3.6 Write the same date, time, and sampler's initials on the label.

3.7 Place the sample container in a ziplock plastic bag and place in a cooler containing ice.

3.8 Fill in appropriate information on the Sample Collection Log Form and the Chain-of-Custody Form.

Example sample labels and tags are attached at the end of this SOP.

## 4.0 ATTACHMENTS

1. Sample Label

## ATTACHMENT 1

## SAMPLE LABEL

| | | |
|---|---|---|
| Tetra Tech NUS, Inc.<br>661 Andersen Drive<br>Pittsburgh, 15220<br>(412)921-7090 | **Project:**<br>**Location:**<br>**CTO:** | |
| **Sample No:** | | **Matrix:** |
| **Date:** | **Time:** | **Preserve:** |
| **Analysis:** | | |
| **Sampled by:** | **Laboratory** | |

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-02

# SAMPLE IDENTIFICATION NOMENCLATURE

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Surface Warfare Center (NSWC). The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

The NSWC Crane Environmental Protection Department must approve any deviations from this procedure.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Pen with indelible ink**
**Sample container labels**

## 3.0 SAMPLE IDENTIFICATION NOMENCLATURE

### 3.1 Monitoring Samples

All monitoring samples collected at NSWC Crane will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number. The sample tracking number will consist of a three- or four-segment alpha-numeric code that identifies the sample's associated solid waste management unit (SWMU) or associated site, sample type, location, and, for aqueous

samples, where applicable, whether a sample is filtered, and/or the sample round number. All sediment samples will be collected at a depth of 0 to 6 inches.

### 3.1.1 Numbering of Samples

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

| NN | AA | NNN | NNNN |
|---|---|---|---|
| SWMU or Site Number | Matrix code | Location number | Sample depth (inches) |

**Character Type:**

A = Alpha

N = Numeric

**SWMU Number (NN):**

17 = PCB Pole Yard

**Matrix code (AA):**

SD = Sediment Sample

SW = Surface Water Sample

**Location number (NNN):**

Sequential number for each media.

**Sample depth (NNNN):**

Listed in inches.

### 3.1.2 Examples of Sample Nomenclature

A surface water sample collected from location 17SW/SD080 at SWMU 17 would be designated as 17SW080.

A sediment sample collected from 6 to 10 inches from location 17SW/SD080 at SWMU 17 would be designated as 17SD0800610.

## 3.2 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the approved field sampling plan and this QAPP Addendum No. 4. They will be designated using a different coding system. The QC code will consist of a four-segment alpha-numeric code that identifies the SWMU or site number, the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

| NN | AA | NNNNNN | NN |
|---|---|---|---|
| SWMU or Site Number | QC Type | Date | Sequence Number (per day) |

The QC types are identified as:

RB = Rinsate Blank (Equipment Blank)
FD = Field Duplicate
SB = Source Water Blank

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

### 3.3.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a surface water sample collected at SWMU 17 on August 3, 2007 would be designated as 17FD08030701.

The third duplicate of the day taken of a sediment sample collected at SWMU 17 on August 17, 2007 would be designated as 17FD08170703.

The first rinsate blank associated with samples collected on October 12, 2007 would be designated as 17RB10120701.

The first source water blank associated with samples collected on October 12, 2007 would be designated as 17SB10120701.

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-03

# SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, and labels are required.

**Site logbook**
**Field logbook**
**Sample label**
**Chain-of-Custody Form**
**Custody seals**
**Equipment calibration log**
**Monitoring Well Inspection Form**
**Water-Level Measurement Form**
**Low-Flow Purge Data Sheet**
**Ground Water Sample Log Sheet**
**Surface Water Sample Log Sheet**

## 3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

## 3.1 Site Logbook

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the field operations leader (FOL).

## 3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is hardbound and paginated.

## 3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

## 3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a multi-part form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL or must include the name of the laboratory in the "Remarks" section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page __ of __" on each COC. The original (top) signed copy of the COC will be placed inside a large Ziploc-type bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Task Order Manager (TOM) or EMAC project manager (PM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

## 3.5 Custody Seal

The custody seal is an adhesive-backed label with a number on each seal. It is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the TtNUS TOM or EMAC PM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.12 Soil and Sediment Sample Log Sheet

The Soil and Sediment Sample Log Sheets are used to document the sampling of soils and sediments (see SOP CTO0020-05).

## 4.0 ATTACHMENTS

1. Chain-of-Custody Record

# ATTACHMENT 1
## CHAIN-OF-CUSTODY RECORD

CHAIN OF CUSTODY | NUMBER | PAGE ____ OF _____

| PROJECT NO: | SITE NAME: | PROJECT MANAGER AND PHONE NUMBER | LABORATORY NAME AND CONTACT: |
|---|---|---|---|
| SAMPLERS (SIGNATURE) | | FIELD OPERATIONS LEADER AND PHONE NUMBER | ADDRESS |
| | | CARRIER/WAYBILL NUMBER | CITY, STATE |

STANDARD TAT ☐
RUSH TAT ☐
☐ 24 hr. ☐ 48 hr. ☐ 72 hr. ☐ 7 day ☐ 14 day

| DATE YEAR | TIME | SAMPLE ID | MATRIX | GRAB (G) COMP (C) | No. OF CONTAINERS | CONTAINER TYPE PLASTIC (P) or GLASS (G) / PRESERVATIVE USED / TYPE OF ANALYSIS | | | | | | | | COMMENTS |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
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|---|---|---|---|---|---|
| 1. RELINQUISHED BY | DATE | TIME | 1. RECEIVED BY | DATE | TIME |
| 2. RELINQUISHED BY | DATE | TIME | 2. RECEIVED BY | DATE | TIME |
| 3. RELINQUISHED BY | DATE | TIME | 3. RECEIVED BY | DATE | TIME |

COMMENTS

DISTRIBUTION: WHITE (ACCOMPANIES SAMPLE) YELLOW (FIELD COPY) PINK (FILE COPY)

3/99
FORM NO. TINUS-001

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-04

# DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations at the NSWC Crane facility.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Waterproof pens**
**Non-latex rubber or plastic gloves**
**Cotton gloves**
**Field logbook**
**Potable water**
**Deionized water**
**LiquiNox detergent**
**Brushes, spray bottles, paper towels, etc.**
**55-gallon drum or other container to collect and transport decontamination fluids**

## 3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has

accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the Liquinox wash.

3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.

3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.

3.7 Document decontamination by recording it in the field logbook.

3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP CTO0020-09 and the QAPP Addendum No. 1.

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-05

# COMPOSITE SAMPLING FOR SEDIMENT

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting composite sediment samples to support the field investigation at solid waste management unit (SWMU) 17 in accordance with the requirements of this QAPP Addendum No. 4.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for composite sediment sampling.

**Disposable medical-grade gloves (e.g., latex, nitrile)**

**Indelible marker**

**Plastic storage bags**

**Shipping containers** (containing ice)

**Disposable plastic trowels or Stainless steel trowels**

**Stainless steel mixing bowls or Ziploc baggies**

**Sample containers:** Sample containers are certified clean by the laboratory supplying the containers.

**Electronic notebook containing:**

- **Sediment Sample Log Forms.**
- **Daily Activity Logs.**
- **Chain-of-Custody Form.**
- **Electronic label maker**

## 3.0 SAMPLING PROCEDURES

Stainless steel or disposable plastic trowels will be employed to collect sediment samples. The sampler will wear clean, disposable, medical-grade gloves.

## 3.1 SEDIMENT COMPOSITE SAMPLING PROCEDURES

3.1.1 Clear the area to be sampled of any surface debris (herbaceous vegetation, twigs, rocks, litter, etc.).

3.1.2 Remove sediment sample from a depth of 0- to 6-inches.

3.1.3 Repeat steps 3.1.1–3.1.2 in the designated sampling area for a specific sample until four discrete sediment sample locations are collected.

3.1.4 Carefully remove the top 6 inches of sediment from the designated sampling area trying not to disturb the underlying sediments. Collect a sample aliquot from a depth of 6 to 12 inches or refusal if less than 12 inches until four discrete sediment sample locations are collected.

3.1.5 Place equal amounts of the sediment for a given sample in a stainless-steel mixing bowl or disposable Ziploc bag and homogenize using a decontaminated stainless steel spoon or dedicated disposable trowel. Place the composite sediment sample into the laboratory supplied sampling container, print, and affix electronically printed label.

3.1.6 For samples that are to be submitted for laboratory analysis, place the labeled sample container into a plastic storage bag and then place the plastic storage bag holding the sample container into a cooler containing ice.

3.1.7 Complete the required information on the Sediment Sample Log Sheet (copy attached at the end of this SOP). Note the location of each aliquot in the "OBSERVATIONS/NOTES" section of the log sheet. Only the notes added within the sample analysis block on the log sheet will be visible on the COC Form for review by laboratory personnel.

3.1.8 Decontaminate the stainless steel trowel and mixing bowl, if employed, in the field between each composite sample location.

## 4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

# ATTACHMENT 1

## SOIL AND SEDIMENT SAMPLE LOG SHEET

### SOIL & SEDIMENT SAMPLE LOG SHEET

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Surface Soil
[] Subsurface Soil
[] Sediment
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**GRAB SAMPLE DATA:**

| Date: | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|
| Time: | | | |
| Method: | | | |
| Monitor Reading (ppm): | | | |

**COMPOSITE SAMPLE DATA:**

| Date: | Time | Depth Interval | Color | Description (Sand, Silt, Clay, Moisture, etc.) |
|---|---|---|---|---|
| | | | | |
| Method: | | | | |
| | | | | |
| Monitor Readings | | | | |
| (Range in ppm): | | | | |
| | | | | |
| | | | | |
| | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Container Requirements | Collected | Other |
|---|---|---|---|
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**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|

**Signature(s):**

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-06

# SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling aqueous samples water soils, and sediments collected at the NSWC Crane.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Shipping labels**

**Custody seals**

**Chain-of-custody (COC) form(s)**

**Sample containers with preservatives**: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.

**Sample shipping containers (coolers)**: All sample shipping containers are supplied by the laboratory.

**Packaging material**: Bubble wrap, ZipLoc® bags, strapping tape, etc.

## 3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at 4°C. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.

3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.

3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a ZipLoc® bag.

3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.

3.6 Put ice into ZipLoc® bags and place a layer of the sealed bags on the bottom of the cooler. Place the sample containers into the shipping cooler on top of the ice in an upright position (containers will be upright, with the exception of the 40-ml vials). Place ZipLoc® bags of ice flat against the sides of the cooler. Continue filling the cooler with samples until the cooler is nearly full and the movement of the sample containers is limited.

3.7 Place a temperature blank in the cooler. Make sure that all samples identified for VOC analysis and their associated trip blank are packed in the same cooler (i.e., VOC samples and trip blank that were stored together during sampling activities can not be separated into different coolers for shipping purposes).

3.8 Add a final layer of ice sealed in ZipLoc® bags to the top of the samples just before the cooler is closed and sealed.

3.9 Place the original (top) signed copy of the COC form inside a large ZipLoc® bag. Tape the bag to the inside of the lid of the shipping cooler that contains the samples for VOC analysis.

3.10 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed, numbered, and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.

3.11 Affix a shipping label to the top of the cooler containing samples for VOC analysis, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.

3.12 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances will sample hold times be exceeded.

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-07

# CALIBRATION AND CARE OF WATER QUALITY METERS

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for the calibration and maintenance of field instruments used to measure water quality and for the proper documentation of calibration and maintenance. The YSI 600-Series Environmental Monitoring System, or a similar system, will be used to measure pH, temperature, oxidation-reduction potential (ORP), specific conductance (SC), dissolved oxygen (DO), and turbidity in water. A LaMotte turbidity meter will be used in conjunction with the water quality meter to measure turbidity. The YSI meter has a multiprobe sensor that can be used in conjunction with a flow-through cell attached to a pump discharge tube to measure water-quality parameters in a ground water discharge or can be immersed in a surface water body such as a stream, pond, or drainage ditch. The LaMotte is a hand held meter that uses a multi-detector optical configuration to assure long term stability and minimize stray light and color interferences.

## 2.0 FIELD FORMS AND EQUIPMENT LIST

The following logbooks, forms, equipment, and supplies are required:

**Site logbook**
**Equipment calibration log sheet**
**YSI Model 600 Serien and Sonde or similar type of meter**: multi-parameter water-quality meter with flow through cell
**LaMotte Turbidity Meter**
**Equipment manual**
**Calibration kit**
**Deionized water, paper towels, spray bottle, etc.**
**Disposable medical-grade gloves (e.g., latex, nitrile)**

## 3.0 PROCEDURES

This section describes the calibration procedures for the YSI Model 600 series and the LaMotte. Each meter is supplied with an instruction manual and will be on site and will be used as the calibration guidance documents. These procedures will list requirements for frequency of calibration and checks to be performed on the meter.

### 3.1 YSI Model 600 Series

The YSI Model 600 series and Sonde is a multi-parameter, water-quality meter that may be used to measure open water bodies (streams, ponds, springs, etc.) with the probe guard installed. With the flow through cell attached, the meter has the ability to measure water-quality parameters in ground water via a pump discharge line. By performing the measurements in the discharge line coming directly from the well, the parameters are measured before the ground water comes in contact with the atmosphere. The parameters measured by the YSI for this field effort are as follows:

- DO
- SC
- Temperature
- pH
- ORP
- Turbidity

#### 3.1.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

#### 3.1.2 Calibration

All the parameters listed in Section 3.0 must be calibrated prior to the start of each field effort. After this initial calibration, the YSI will be checked each day that it is used. If the check shows any out-of-specification readings, the specific probe will be recalibrated. Meter specifications can be found in the

equipment manual, starting on page 248. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration buffers and standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

### 3.1.3 Tips for Good Calibration

- The DO calibration is a water-saturated air calibration. Make certain to loosen the calibration cup seal to allow pressure to equilibrate before calibrating.
- Make certain that sensors are completely submersed in solution and readings are stable when calibration values are entered.
- Use a small amount of calibration solution (previously used solution may be used, then discarded for this purpose) to pre-rinse the sonde.
- Fill a bucket with ambient temperature water to rinse the sonde between calibration solutions.
- Make sure to rinse and dry the probe between calibration solutions. This will reduce carry-over contamination and increase the accuracy of the calibration.

## 3.2 Lamotte Turbidity Meter

The Lamotte turbidity meter is a hand held meter that measures the amount of suspended matter in water using the Nephelometric method.

### 3.2.1 Documentation

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including the frequency of calibration, type of standards used, and checks performed on calibration during the course of using the equipment. An Equipment Calibration Log must be maintained for each measuring device that requires calibration. Entries must be made for each day the equipment is used. A blank Equipment Calibration Log form is attached at the end of this SOP.

### 3.2.2 Calibration

Turbidity must be calibrated prior to the start of each field effort. After this initial calibration, the LaMotte will be calibrated each day that it is used. If the check shows any out-of-specification readings, the meter will be recalibrated. Meter specifications can be found in the equipment manual. Calibration and calibration checks will be documented in the field logbook and on the Equipment Calibration Log. The name, lot number, and expiration date for all calibration standards used will be recorded on the Equipment Calibration Log. The meter's model, serial number, and name of rental company will also be recorded on the equipment calibration form.

### 3.2.2 Tips for Good Calibration

- Thoroughly clean the standard vial with a chem wipe to remove finger prints.
- Make sure that the vial is properly aligned according the manual recommendations.

## 4.0 MAINTENANCE

The YSI Meter and LaMotte will be rented for the duration of each brief field effort. Therefore, little field maintenance will be required. For any maintenance other than the routine cleaning, calibrating, or battery charging, the instrument should be returned to the vendor and a replacement sent immediately to the job site.

### 4.1 Meter Storage for the YSI

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. During these breaks, the meter will be charged. One-half inch of tap or distilled water will be placed in the meter calibration cup and the cup threaded onto the sonde. The key for short-term storage of probes is to use a minimal amount of water so the calibration cup will remain at 100 percent humidity. The water level must be low enough so that none of the probes are actually immersed. Proper storage of the sonde between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

**Multi-parameter short term storage key points:**

- Use enough water to provide humidity but not enough to cover the probe surfaces.

- Make sure the storage vessel is sealed to minimize evaporation.
- Check periodically to make certain that water is still present.

### 4.2 Probe Cleaning

- Rinse the probe thoroughly with potable water.
- Wash the probe in a mild solution of Liquinox and water and wipe with paper towels and/or cotton swabs.
- Rinse and soak the probe in deionized water.
- If stronger cleaning is required, consult Section 2.10 on page 89 of the equipment manual.

Note: Reagents that are used to calibrate and check the YSI may be hazardous. Review the health and safety plan, Appendix A of the equipment manual, and Material Safety Data Sheets (MSDSs), all of which are on file in the field trailer.

### 4.3 Meter Storage for the LaMotte

For this field effort, the meter storage will be short term, [i.e. over night or between work shifts (4-day break)]. Proper storage of the meter between usage will extend its life and will also ensure that the unit is ready for use as quickly as possible for the next application.

**Short term storage key points:**

- Make sure the storage vessel is moisture free and sealed.

### 4.4 Sample Vial Cleaning

- Rinse the vial thoroughly with potable water to remove sediments.
- Wipe with chem.-wipes or cotton swabs.

## 5.0 ATTACHMENTS

1. Documentation of Field Calibration
2. Equipment Calibration Log

# ATTACHMENT 1
## DOCUMENTATION OF FIELD CALIBRATION

Tetra Tech NUS, Inc.

**DOCUMENTATION OF FIELD CALIBRATION**

PROJECT NAME: _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

| Date of Calibration | Person Performing Calibration | INITIAL READINGS | | | | | | FINAL READINGS | | | | | | Calibration Standard (Lot No.) | Remarks and Comments |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | PH | COND | TURB | DO | TEMP | SAL | PH | COND | TURB | DO | TEMP | SAL | | |
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# ATTACHMENT 2
# EQUIPMENT CALIBRATION LOG

Tetra Tech NUS, Inc.

## EQUIPMENT CALIBRATION LOG

PROJECT NAME : _____ INSTRUMENT NAME/MODEL: _____

SITE NAME: _____ MANUFACTURER: _____

PROJECT No.: _____ SERIAL NUMBER: _____

| Date of Calibration | Instrument I.D. Number | Person Performing Calibration | Instrument Settings | | Instrument Readings | | Calibration Standard (Lot No.) | Remarks and Comments |
|---|---|---|---|---|---|---|---|---|
| | | | Pre-calibration | Post-calibration | Pre-calibration | Post-calibration | | |
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# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-08

# SURFACE WATER SAMPLING

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for collecting surface water samples at the NSWC Crane facility.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Surface Water Sample Log Sheet**: A copy of this form is attached at the end of this SOP.

**Field logbook**

**Writing utensil**

**Multi-parameter water-quality meter**: The water-quality meter is used for the measurement of dissolved oxygen, pH, specific conductance, temperature, oxidation-reduction potential, and turbidity (see SOP CTO0020-07).

**LaMotte Turbidity Meter:** Used to measure turbidity in the field (see SOP CTO0020-07).

**Disposable sample containers**: Disposable sample containers are used to fill sample containers and transport sample(s) to a pump for filtering.

**Labeled sample containers**: Prelabeled, certified-clean sample containers will be provided by the laboratory that performs the analyses.

**Sample tags**: One tag is to be completed and attached to each sample container.

**0.45-micron filter assembly**: These are single-use filter cartridges used to filter samples scheduled for dissolved metals or dissolved thorium isotope analyses. The filters become investigation-derived waste (IDW) after one use.

**Peristaltic pump**

**Silicon tubing**

**Ziploc-type plastic storage bags**

**Shipping containers (coolers)**

**Trip blank sample** (if VOC samples are being collected)

**Temperature blank**

## 3.0 SURFACE WATER SAMPLING PROCEDURES

3.1 The same methods will be used to collect surface water and seep samples. Sampling will start at the downstream end of a stream and proceed to the farthest upstream location.

3.2 While standing downstream or from the bank, gently remove any floating leaves or twigs that may be present in a sample pool area in a manner that will not disturb the bottom sediment.

3.3 Obtain measurements of dissolved oxygen, pH, specific conductance, temperature, turbidity, and oxidation-reduction potential immediately downstream of the sample collection point using the multi-parameter water-quality meter and LaMotte Turbidity Meter (see SOP CTO0020-11). Record the readings in the appropriate fields on the Surface Water Sample Log Sheet.

3.4 For sample locations having a turbidity of 10 Nephelometric Turbidity Units (NTUs) or less, while standing downstream or from the bank, place the sample container in the water at the sampling location at a 45-degree angle and lower it to approximately half the sample pool depth. With the mouth of the container facing upstream, fill the container with water, being careful not to disturb the sediment. For sample locations having a turbidity greater than 10 NTUs, the sample will first be passed through a 0.45-micron filter prior to filling the sample container.

3.5 All samples will be collected into certified-clean, non-preserved bottles supplied by the laboratory performing the analyses. Sample containers should <u>not</u> be filled completely; a small amount of air should be left at the top.

3.6 Record the date and time that the sample containers are filled on the Surface Water Sample Log Sheet, the sample labels, and the Chain-of-Custody Form.

3.7 After the sample label is complete and checked, place the sample container into a ziploc-type plastic storage bag and place the plastic storage bag holding the sample container into a cooler containing ice.

3.8 Repeat steps 3.3 through 3.6 until all the sample bottles containing unfiltered samples have been filled.

3.9 Estimate the flow rate of the stream or spring in accordance with SOP CTO0020-10. This is an estimate only. Round the flow rate to the nearest 5 gallons and record this number on the Surface Water Sample Log Sheet.

3.10 Decontaminate all equipment and load the equipment and the sample cooler in the sample vehicle for transport.

## 4.0 ATTACHMENTS

1. Surface Water Sample Log Sheet

## ATTACHMENT 1

## SURFACE WATER SAMPLE LOG SHEET

Tetra Tech NUS, Inc. **SURFACE WATER SAMPLE LOG SHEET**

Page___ of ___

Project Site Name: _____
Project No.: _____

[] Stream
[] Spring
[] Pond
[] Lake
[] Other: _____
[] QA Sample Type: _____

Sample ID No.: _____
Sample Location: _____
Sampled By: _____
C.O.C. No.: _____

Type of Sample:
[] Low Concentration
[] High Concentration

**SAMPLING DATA:**

| Date:<br>Time: | Color<br>Visual | pH<br>Standard | S.C.<br>mS/cm | Temp.<br>Degrees C | Turbidity<br>NTU | DO<br>mg/l | Salinity<br>% | ORP<br>mV |
|---|---|---|---|---|---|---|---|---|
| Depth:<br>Method: | | | | | | | | |

**SAMPLE COLLECTION INFORMATION:**

| Analysis | Preservative | Container Requirements | Collected |
|---|---|---|---|
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**OBSERVATIONS / NOTES:**

**MAP:**

**Circle if Applicable:**

| MS/MSD | Duplicate ID No.: |
|---|---|
| | |

**Signature(s):**

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-09

# MANAGEMENT OF INVESTIGATION-DERIVED WASTE

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at the NSWC Crane facility. The following types of IDW will be generated during this investigation:

- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Health and safety equipment (with PPE)**
**Decontamination equipment**
**Field logbook and indelible ink pen**
**Plastic sheeting and/or tarps**
**55-gallon drums with sealable lids**
**IDW labels for drums**
**Wastewater container tanks**
**Plastic garbage bags**

## 3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities.

## 3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include SWMU 8 pond water and decontamination solutions from excavation and sampling equipment. These wastes will be collected and managed in accordance with the IMWPs (TtNUS, 2006 a through 2006d).

## 3.2 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NSWC Crane.

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-10

# ESTIMATING FLOW IN SMALL STREAMS

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) provides technical guidance on the measurement of stream channel cross-section and flow in a small stream. Stream flow measurements will be obtained to evaluate the migration potential of contaminants in a stream channel. This method is applicable for free-flowing streams with uniform flow and widths greater than 3 feet and depths greater than about 6 inches. Stream flow rates will be measured at the same time as water-level measurements are collected.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

**Measuring rod or yardstick**
**Measuring tape**
**Waders or rubber boots**
**Colored flagging tape**
**2x2 inch wooden stakes**
**Bound field logbook**
**Waterproof pen**
**Orange, apple, or small piece of wood**
**Surface Water Sample Log Sheet**
**Indelible marker**
**Stopwatch**

## 3.0 PROCEDURES

3.1 Establish a transect across the stream, along a straight reach where the stream is relatively narrow and velocity across the stream is relatively uniform. Orient the transect perpendicular to the flow.

3.2 Mark both ends of the transect with colored plastic flagging tape. Tie the flagging to a tree branch or other elevated (about eye level) object. Drive wooden stakes into the ground at both ends of

transect and tack a short piece of brightly colored flagging to the stakes. Use a black marker to identify the transect number, which will be the same as the surface water sample location.

3.3 Measure the total distance across the stream with a measuring tape and record the stream width (in feet) in the field logbook.

3.4 Measure the depth of water in the stream at three or four points across the stream. Record these measurements and calculate an average depth. Record the average depth (in feet) in the field logbook.

3.5 Measure a distance of 20 feet upstream of the transect. Tie a piece of flagging on a tree branch or other visible location above the stream.

3.6 Go another 20 feet upstream and place the orange or apple in the center of the stream or where the water current is fastest.

3.7 Using a watch with a timer or a stopwatch, measure the length of time it takes the orange (or apple) to travel from the first marker flag to the transect location (distance of 20 feet). Record the time.

3.8 Repeat step 3.7 two more times. Record the readings and calculate an average time.

3.9 Calculate approximate flow rate in the stream channel by multiplying the stream width (from step 3.3) times the average depth (from step 3.4) times the average velocity (from step 3.8). Record the estimated flow rate (in $ft^3/s$) in the field logbook and on the Surface Water Sample Collection Log Sheet.

# STANDARD OPERATING PROCEDURE
# NUMBER CTO0020-11

# MEASUREMENT OF WATER QUALITY PARAMETERS

## 1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for measurement of water quality parameters during the collection of aqueous samples to support the field investigation at solid waste management unit (SWMU) 17 in accordance with the requirements of this QAPP Addendum No. 4.

## 2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following field forms and equipment are required for measuring water quality parameters during aqueous sampling:

**Disposable medical-grade gloves (e.g., latex, nitrile)**

**Indelible marker**

**Plastic storage bags**

**Multi-Parameter Water Quality Meter**

**Turbidity Meter**

**Shipping containers** (containing ice)

**Sample containers:** Sample containers are certified clean by the laboratory supplying the containers.

**Electronic notebook containing:**

- **Surface Water Sample Log Forms.**
- **Daily Activity Logs.**
- **Chain-of-Custody Form.**
- **Electronic label maker**

## 3.0 GLOSSARY

Conductivity – Conductivity is a numerical expression of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions, their total concentration, mobility, valence, and relative concentrations, and on temperature of measure. Conductivity is highly dependent on temperature and should be reported at a particular temperature, i.e., 20.2 mS/cm at 14°C.

Dissolved Oxygen (DO) – DO levels in natural and wastewater depend on the physical, chemical, and biochemical activities in the water sample.

Oxidation-Reduction Potential (ORP) - A measure of the activity ratio of oxidizing and reducing species as determined by the electromotive force developed by a noble metal electrode, immersed in water, as referenced against a standard hydrogen electrode.

pH - The negative logarithm (base 10) of the hydrogen ion activity. The hydrogen ion activity is related to the hydrogen ion concentration, and, in a relatively weak solution, the two are nearly equal. Thus, for all practical purposes, pH is a measure of the hydrogen ion concentration.

pH Paper - Indicator paper that turns different colors depending on the pH of the solution to which it is exposed. Comparison with color standards supplied by the manufacturer will then give an indication of the solution's pH.

Turbidity – Turbidity in water is caused by suspended matter, such as clay, silt, fine organic and inorganic matter. Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample.

## 4.0 ON-SITE WATER QUALITY TESTING

This section describes the procedures and equipment required to measure the following parameters of an aqueous sample in the field:

- pH
- Specific Conductance
- Temperature
- Dissolved Oxygen (DO)
- Oxidation-Reduction Potential (ORP)
- Turbidity

This section also provides general information for measuring the parameters listed above with instruments and techniques in common use. Since instruments from different manufacturers may vary, review of the manufacturer's literature pertaining to the use of a specific instrument is required before use. Most meters used to measure field parameters require calibration on a daily basis. Refer to SOP_CTO0020-07 for example equipment calibration log.

## 4.1 Measurement of pH

### 4.1.1 General

Measurement of pH is one of the most important and frequently used tests in water chemistry. Practically every phase of water supply and wastewater treatment such as acid-base neutralization, water softening, and corrosion control is pH dependent. Likewise, the pH of leachate can be correlated with other chemical analyses to determine the probable source of contamination. It is therefore important that reasonably accurate pH measurements be taken.

Two methods are given for pH measurement: the pH meter and pH indicator paper. The indicator paper is used when only a rough estimate of the pH is required, and the pH meter when a more accurate measurement is needed. The response of a pH meter can be affected to a slight degree by high levels of colloidal or suspended solids, but the effect is usually small and generally of little significance. Consequently, specific methods to overcome this interference are not described. The response of pH paper is unaffected by solution interferences from color, turbidity, colloidal or suspended materials unless extremely high levels capable of coating or masking the paper are encountered. In such cases, use of a pH meter is recommended.

### 4.1.2 Principles of Equipment Operation

Use of pH papers for pH measurement relies on a chemical reaction caused by the acidity or alkalinity of the solution created by the addition of the water sample reacting with the indicator compound on the paper. Various types of pH papers are available, including litmus (for general acidity or alkalinity determination) and specific pH range hydrion paper.

Use of a pH meter relies on the same principle as other ion-specific electrodes. Measurement relies on establishment of a potential difference across a glass or other type of membrane in response to (in this instance, hydrogen) ion concentration across that membrane. The membrane is conductive to ionic species and, in combination with a standard or reference electrode, a potential difference proportional to the ion concentration is generated and measured.

### 4.1.3 Equipment

The following equipment is needed for taking pH measurements:

- Stand-alone portable pH meter, or combination meter (e.g., Horiba U-10), or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

- Combination electrode with polymer body to fit the above meter (alternately a pH electrode and a reference electrode can be used if the pH meter is equipped with suitable electrode inputs).

- Buffer solutions, as specified by the manufacturer.

- pH indicator paper, to cover the pH range 2 through 12.

- Manufacturer's operation manual.

4.1.4 Measurement Techniques for Field Determination of pH

pH Meter

The following procedure is used for measuring pH with a pH meter (meter standardization is according to manufacturer's instructions):

- Inspect the instrument and batteries prior to initiation of the field effort.

- Check the integrity of the buffer solutions used for field calibration. Buffer solutions need to be changed often as a result of degradation upon exposure to the atmosphere.

- If applicable, make sure all electrolyte solutions within the electrode(s) are at their proper levels and that no air bubbles are present within the electrode(s).

- Calibrate on a daily use basis (or as recommended by manufacturer) following manufacturer's instructions. Record calibration data on an equipment calibration log sheet (See SOP_CTO0020-07).

- Immerse the electrode(s) in the sample or body of water. Stabilization may take several seconds to minutes. If the pH continues to drift, the sample temperature may not be stable, a physical reaction (e.g., degassing) may be taking place in the sample, or the meter or electrode may be malfunctioning. This must be clearly noted in the logbook.

- Read and record the pH of the sample. pH shall be recorded to the nearest 0.01 pH unit. Also record the sample temperature.

- Rinse the electrode(s) with deionized water.

- Store the electrode(s) in an appropriate manner when not in use.

Any visual observation of conditions which may interfere with pH measurement, such as oily materials, or turbidity, shall be noted.

pH Paper

Use of pH paper is very simple and requires no sample preparation, standardization, etc. pH paper is available in several ranges, including wide-range (indicating approximately pH 1 to 12), mid-range (approximately pH 0 to 6, 6 to 9, 8 to 14) and narrow-range (many available, with ranges as narrow as 1.5 pH units). The appropriate range of pH paper shall be selected. If the pH is unknown the investigation shall start with wide-range paper and proceed with successively narrower range paper until the sample pH is adequately determined.

## 4.2 Measurement of Specific Conductance

### 4.2.1 General

Conductance provides a measure of dissolved ionic species in water and can be used to identify the direction and extent of migration of contaminants in groundwater or surface water. It can also be used as a measure of subsurface biodegradation or to indicate alternate sources of contamination.

Conductivity is a numerical expression of the ability of a water sample to carry an electric current. This value depends on the total concentration of the ionized substances dissolved in the water and the temperature at which the measurement is made. The mobility of each of the various dissolved ions, their valences, and their actual and relative concentrations affect conductivity.

Most conductivity meters in use today display specific conductance (SC); units of milliSiemens per centimeter, which is the conductivity normalized to temperature @ 25°C. This format (SC) is the required units recorded on the sample log field form.

### 4.2.2 Principles of Equipment Operation

An aqueous system containing ions will conduct an electric current. In a direct-current field, the positive ions migrate toward the negative electrode, while the negatively charged ions migrate toward the positive electrode. Most inorganic acids, bases and salts (such as hydrochloric acid, sodium carbonate, or sodium chloride, respectively) are relatively good conductors. Conversely, organic compounds such as sucrose or benzene, which do not dissociate in aqueous solution, conduct a current very poorly, if at all.

A conductance cell and a Wheatstone Bridge (for the measurement of potential difference) may be used for measurement of electrical resistance. The ratio of current applied to voltage across the cell may also be used as a measure of conductance. The core element of the apparatus is the conductivity cell containing the solution of interest. Depending on ionic strength of the aqueous solution to be tested, a potential difference is developed across the cell which can be converted directly or indirectly (depending on instrument type) to a measurement of specific conductance.

### 4.2.3 Equipment

The following equipment is needed for taking specific conductance (SC) measurements:

- Stand alone portable conductivity meter, or combination meter (e.g., Horiba U-10), or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Calibration solution, as specified by the manufacturer.
- Manufacturer's operation manual.

A variety of conductivity meters are available which may also be used to monitor salinity and temperature. Probe types and cable lengths vary, so equipment must be obtained to meet the specific requirement of the sampling program.

### 4.2.4 Measurement Techniques for Specific Conductance

The steps involved in taking specific conductance measurements are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.

- Calibrate on a daily use basis (or as recommended by manufacturer), according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet. Potassium chloride solutions with a SC closest to the values expected in the field shall be used for calibration.

- Rinse the cell with one or more portions of the sample to be tested or with deionized water.

- Immerse the electrode in the water and measure the conductivity.

- Read and record the results in a field logbook or sample log sheet.

- Rinse the electrode with deionized water.

If the specific conductance measurements become erratic, recalibrate the instrument and see the manufacturer's instructions for details.

## 4.3 Measurement of Temperature

### 4.3.1 General

In combination with other parameters, temperature can be a useful indicator of the likelihood of biological action in a water sample. It can also be used to trace the flow direction of contamination. Temperature measurements shall be taken in-situ, or as quickly as possible in the field. Collected water samples may rapidly equilibrate with the temperature of their surroundings.

### 4.3.2 Equipment

Temperature measurements may be taken with alcohol-toluene, mercury filled, dial-type thermometers or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22). In addition, various meters such as specific conductance or dissolved oxygen meters, which have temperature measurement capabilities, may also be used. Using such instrumentation along with suitable probes and cables, in-situ measurements of temperature at great depths can be performed.

### 4.3.3 Measurement Techniques for Water Temperature

If a thermometer is used to determine the temperature for a water sample:

- Immerse the thermometer in the water until temperature equilibrium is obtained (1-3 minutes).
- Record values in a field logbook or sample log sheet.

If a temperature meter or probe is used, the instrument shall be calibrated according to manufacturer's recommendations.

## 4.4 Measurement of Dissolved Oxygen

### 4.4.1 General

Dissolved oxygen (DO) levels in natural water and wastewater depend on the physical, chemical and biochemical activities in the water body. Conversely, the growth of many aquatic organisms as well as the rate of corrosivity, are dependent on the dissolved oxygen concentration. Thus, analysis for dissolved oxygen is a key test in water pollution and waste treatment process control. If at all possible, DO measurements shall be taken in-situ, since concentration may show a large change in a short time if the sample is not adequately preserved.

The monitoring method discussed herein is limited to the use of dissolved oxygen meters only. Chemical methods of analysis (i.e., Winkler methods) are available, but require more equipment and greater sample manipulation. Furthermore, DO meters, using a membrane electrode, are suitable for highly polluted waters, because the probe is completely submersible, and is not susceptible to interference caused by color, turbidity, colloidal material or suspended matter.

### 4.4.2 Principles of Equipment Operation

Dissolved oxygen probes are normally electrochemical cells that have two solid metal electrodes of different nobility immersed in an electrolyte. The electrolyte is retained by an oxygen-permeable membrane. The metal of highest nobility (the cathode) is positioned at the membrane. When a suitable potential exists between the two metals, reduction of oxygen to hydroxide ion ($OH^-$) occurs at the cathode surface. An electrical current is developed that is directly proportional to the rate of arrival of oxygen molecules at the cathode.

Since the current produced in the probe is directly proportional to the rate of arrival of oxygen at the cathode, it is important that a fresh supply of sample always be in contact with the membrane. Otherwise, the oxygen in the aqueous layer along the membrane is quickly depleted and false low readings are obtained. It is therefore necessary to stir the sample (or the probe) constantly to maintain fresh solution near the membrane interface. Stirring, however, shall not be so vigorous that additional oxygen is

introduced through the air-water interface at the sample surface. To avoid this possibility, some probes are equipped with stirrers to agitate the solution near the probe, while leaving the surface of the solution undisturbed.

Dissolved oxygen probes are relatively unaffected by interferences. Interferences that can occur are reactions with oxidizing gases (such as chlorine) or with gases such as hydrogen sulfide, which are not easily depolarized from the indicating electrode. If a gaseous interference is suspected, it shall be noted in the field log book and checked if possible. Temperature variations can also cause interference because probes exhibit temperature sensitivity. Automatic temperature compensation is normally provided by the manufacturer.

### 4.4.3 Equipment

The following equipment is needed to measure dissolved oxygen concentration:

- Stand alone portable dissolved oxygen meter, or combination meter (e.g., Horiba U-10), or combination meters equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Sufficient cable to allow the probe to contact the sample.
- Manufacturer's operation manual.

### 4.4.4 Measurement Techniques for Dissolved Oxygen Determination

Probes differ as to specifics of use. Follow the manufacturer's instructions to obtain an accurate reading. The following general steps shall be used to measure the dissolved oxygen concentration:

- The equipment shall be calibrated and have its batteries checked before going to the field.

- The probe shall be conditioned in a water sample for as long a period as practical before use in the field. Long periods of dry storage followed by short periods of use in the field may result in inaccurate readings.

- The instrument shall be calibrated in the field according to manufacturer's recommendations or in a freshly air-saturated water sample of known temperature.

- Record all pertinent information on an equipment calibration sheet.

- Rinse the probe with deionized water.
- Immerse the probe in the water.
- Record the dissolved oxygen content and temperature of the sample in a field logbook or sample log sheet.
- Rinse the probe with deionized water.
- Recalibrate the probe when the membrane is replaced, or as needed. Follow the manufacturer's instructions.

Note that in-situ placement of the probe is preferable, since sample handling is not involved. This however, may not always be practical.

Special care shall be taken during sample collection to avoid turbulence which can lead to increased oxygen solubilization and positive test interferences.

## 4.5 Measurement of Oxidation-Reduction Potential

### 4.5.1 General

The oxidation-reduction potential (ORP) provides a measure of the tendency of organic or inorganic compounds to exist in an oxidized state. The ORP parameter therefore provides evidence of the likelihood of anaerobic degradation of biodegradable organics or the ratio of activities of oxidized to reduced species in the sample.

### 4.5.2 Principles of Equipment Operation

When an inert metal electrode, such as platinum, is immersed in a solution, a potential is developed at that electrode depending on the ions present in the solution. If a reference electrode is placed in the same solution, an ORP electrode pair is established. This electrode pair allows the potential difference between the two electrodes to be measured and is dependent on the concentration of the ions in solution. By this measurement, the ability to oxidize or reduce species in solution may be determined. Supplemental measurements, such as dissolved oxygen, may be correlated with ORP to provide a knowledge of the quality of the solution, water, or wastewater.

4.5.3 Equipment

The following equipment is needed for measuring the oxidation-reduction potential of a solution:

- Combination meters with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).
- Reference solution as specified by the manufacturer.
- Manufacturer's operation manual.

4.5.4 Measurement Techniques for Oxidation-Reduction Potential

The following procedure is used for measuring oxidation-reduction potential:

- The equipment shall be checked using the manufacturer's recommended reference solution and have its batteries checked before going to the field.

- Thoroughly rinse the electrode with deionized water.

- If the probe does not respond properly to the recommended reference solution, then verify the sensitivity of the electrodes by noting the change in millivolt reading when the pH of a test solution is altered. The ORP will increase when the pH of a test solution decreases, and the ORP will decrease if the test solution pH is increased. Place the sample in a clean container and agitate the sample. Insert the electrodes and note the ORP drops sharply when the caustic is added (i.e., pH is raised) thus indicating the electrodes are sensitive and operating properly. If the ORP increases sharply when the caustic is added, the polarity is reversed and must be corrected in accordance with the manufacturer's instructions or the probe should be replaced.

- Record all pertinent information on an equipment calibration log sheet.

## 4.6 Measurement of Turbidity

4.6.1 General

Turbidity is an expression of the optical property that causes light to be scattered and absorbed rather than transmitted in a straight line through the sample. Turbidity in water is caused by suspended matter, such as clay, silt, finely divided organic and inorganic matter, soluble colored organic compounds, and microscopic organisms, including plankton.

It is important to obtain a turbidity reading immediately after taking a sample, since irreversible changes in turbidity may occur if the sample is stored too long.

### 4.6.2 Principles of Equipment Operation

Turbidity is measured by the Nephelometric Method. This method is based on a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension under the same conditions. The higher the scattered light intensity, the higher the turbidity.

Formazin polymer is used as the reference turbidity standard suspension because of its ease of preparation combined with a higher reproducibility of its light-scattering properties than clay or turbid natural water. The turbidity of a specified concentration of formazin suspension is defined as 40 nephelometric units. This same suspension has an approximate turbidity of 40 Jackson units when measured on the candle turbidmeter. Therefore, nephelometric turbidity units (NTU) based on the formazin preparation will approximate units derived from the candle turbidimeter but will not be identical to them.

### 4.6.3 Equipment

The following equipment is needed for turbidity measurement:

- Light meter (e.g., LaMotte 2020) which calibrates easily using test cells with standards of 0.0 NTUs, and 10 NTUs, or combination meter (e.g., Horiba U-10), or combination meter equipped with an in-line sample chamber (e.g., YSI 600 series and Horiba U-22).

- Calibration solution, as specified by the manufacturer.

- Manufacturer's operation manual.

### 4.6.4 Measurement Techniques for Turbidity

The steps involved in taking turbidity measurements utilizing an electrode (e) or light meter (l) are listed below (standardization is according to manufacturer's instructions):

- Check batteries and calibrate instrument before going into the field.

- Check the expiration date (etc.) of the solutions used for field calibration.

- Calibrate on a daily use basis, according to the manufacturer's instructions and record all pertinent information on an equipment calibration log sheet.

- Rinse the electrode with one or more portions of the sample to be tested or with deionized water (applies to "e").

- Fill the light meters glass test cell with ~5 ml of sample, screw on cap, wipe off glass, place test cell in light meter and close the lid (applies to "l").

- Immerse the electrode in the sample and measure the turbidity (applies to "e").

- The reading must be taken immediately as suspended solids will settle over time resulting in a lower, inaccurate turbidity reading.

- Read and record the results in a field logbook or sample log sheet. Include a physical description of the sample, including color, qualitative estimate of turbidity, etc.

- Rinse the electrode or test cell with deionized water.

# APPENDIX B

# LABORATORY SOPs

# LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
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| LTL-1001 | 8 | Elements of SOP and Method Formats |
| LTL-1002 | 8 | Document Tracking and Control |
| LTL-1003 | 5 | Chain-of-Custody and Documentation Procedures |
| LTL-1004 | 6 | Documentation of Analyst Competence and Training |
| LTL-1005 | 8 | Analytical Balances |
| LTL-1006 | 6 | Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance |
| LTL-1007 | 6 | Use of Instrument Records and Logbooks |
| LTL-1008 | 8 | QC Corrective Action |
| LTL-1009 | 4 | Blind Spike Program |
| LTL-1011 | 8 | Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits |
| LTL-1012 | 4 | Solvent QC Monitoring for Trace Residue Analysis |
| LTL-1013 | 7 | Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials |
| LTL-1017 | 3 | Internal Audit Procedures |
| LTL-1018 | 4 | Overview of Review and Approval Practices for Validatable Data Packages |
| LTL-1019 | 4 | Controlling, Maintaining, and Monitoring Laboratory Logbooks |
| LTL-1020 | 3 | Integration of IC, GC, HPLC and GCMS Peaks |
| LTL-2001 | 9 | Waste Segregation and Disposal |
| LTL-3205 | 0 | Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081B/8082A by 3535A-SPE) |
| LTL-3303 | 5 | Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Soil (8081B/8082A by 3545A) |
| LTL-4002 | 10 | Electronic Sample Entry and Log-In |
| LTL-4201 | 0 | Package Deliverables for all Reporting Levels |

# LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
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| SOP NUMBER | REV # | TITLE |
|---|---|---|
| LTL-7003 | 4 | Inorganic Glass Cleaning Procedure |
| LTL-7015 | 5 | Acid Digestion of Sediments, Sludges, and Soils Using SW846 Method 3050B |
| LTL-8000 | 5 | Determination of Retention Time Windows |
| LTL-8084 | 9 | Analysis of Organochlorine Pesticides and PCBs by SW 846 Methods 8081B and 8082A |

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1001

Title: **Elements of SOP and Method Formats**

Revision history:

| Number | Date |
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| 5 | 6/25/02 |
| 4 | 5/3/99 |
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Revised by: Harry Romberg Date: 4/26/07
Harry Romberg, QA Officer

Approved by: Hugh Prentice Date: 4/26/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to define the process of creating SOPs and Methods.

1.2 Scope

1.2.1 SOPs are considered to be administrative and other non-analytical tasks.

1.2.2 Methods are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. This document assumes that the user either has some knowledge of the word processor being used or can figure out how to perform the basic operations necessary.

1.2.3 This SOP does not address document control except as it relates to numbering of the SOP. Document control is addressed in Laucks SOP LTL-1002.

## 2. Procedures

2.1 Word Processing Format

2.1.1 All new SOPs are written in WORD 95 format or higher. Older SOPs may have originally been written in earlier versions of WORD and still have some remnants of that version which can cause unintended formatting or other issues but most such discrepancies have been resolved. Although the older XyWrite has long been discontinued and documents not written in this format for years, remnants may continue to exist. If any of these formats are encountered, they should be updated to meet the current format.

2.1.2 Flow charts may either be photocopied from methods, scanned (usually as TIFF files) and inserted into the document electronically or prepared from programs such as Flow Charting PDQ (Patton & Patton Software) and inserted into the document electronically. Any of these electronic files should be kept together with the electronic version of the document to facilitate its modification and inclusion into future updates. Where possible, it is preferable to actually insert the file directly into the electronic document so that it will not be forgotten or lost as files are moved or copies made.

2.2 Initiating an SOP or Method

2.2.1 Prior to creating a new SOP or Method or revising an existing document, the prospective author or supervisor should first complete a Document Control Form as specified in the document control SOP, LTL-1002. This form can be obtained from the QA Department

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or can be found amongst the document templates in WORD (Y:\template_QA\Doc_cont_form.dot). If the user has Y:\template as their workgroup template file location, they only need to select [File][New][More…][QA][Doc_cont_form].

2.2.2 "SOPs" refer to be administrative and other non-analytical tasks. "Methods" are considered to be technical analytical procedures and are generally derived from EPA or other analysis methods. For tracking and control purposes, all will be assigned a number by the QA Officer or designee. This number will begin with the letters LTL- (such as this SOP, LTL-1001). A number should be obtained from QA before the author begins writing the SOP but if this is not possible, a number must be obtained before the SOP can be turned in for review.

2.2.3 If a revision of a previous document is being undertaken, the SOP or Method number will remain the same but the revision number will be incremented. Revision numbers of new documents will automatically be assigned as 0 with subsequent revisions being incremented by 1 (1, 2, 3, etc.).

2.2.4 For new SOPs, the author may then use the appropriate Word template (SOPhead, Iorgtemp or Orgtemp) from the [File][New][More…][QA] tab presented in WORD (if the template folder points to the same directory noted above in 2.2.1). Hardcopies of these formats are not included in this SOP but may be accessed by the reader using the above means. These formats may change somewhat without updating of this SOP but if the author accesses the template from the laboratory network in this manner, the latest version will be automatically used.

2.2.5 These are meant for guidance only and changes to the formats will be allowed if they present a more complete and accurate account of method performance. While it is entirely up to the author to change any part of one of these templates to suit the specific procedure, these elements will be looked at in the review process and must be included unless they are inappropriate to the procedure being described.

2.2.5.1 **SOPhead** is a general SOP template. This template is NOT to be used for analytical methods as it does not contain all of the necessary elements of a method (i.e. QC requirements). Specific elements of a method are outlined below and in the method templates Iorgtemp and Orgtemp.

2.2.5.2 **Orgtemp** is the method template which has been created for chromatographic analytical methods. It is primarily written for organic analysis but may also be applied to such inorganic techniques as ion chromatography.

2.2.5.3 **Iorgtemp** is for most non-chromatographic methods, which comprises most inorganic analyses.

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2.2.6 The template should be opened and appropriate information filled in. Most of the items which need input are highlighted in red in the template. This does not mean that text which is black cannot be modified or even deleted if it is not pertinent to the analysis in question. It also enables QA to maintain historical versions of SOPs.

2.2.7 Draft versions of SOPs **MUST** be worked on in the "projects" drive (p: on most computers). They should be located in the p:\sop directory under the subdirectory most relevant to the areas addressed by the SOP. For instance, in writing this SOP, it was stored in p:\sop\qa_sops. Metals SOPs would be worked on in the p:\sop\metals subdirectory. QA will transfer the final document to the appropriate location for permanent storage and archival in order to maintain electronic copies of all of the appropriate revisions. This is both for historical record and so that subsequent revisions can be made on the existing controlled SOP rather than a copy of unknown origin or content that may not be exactly the same as the written version.

2.3 Revising an SOP

2.3.1 QA will transfer the last revision of the SOP to P:\SOP\[*department*] where the author will make whatever changes are considered necessary. Note, the file will now be either a .doc file or given the extension .R00, .R01, .R02....(depending on the revision), rather than a blank template. As noted earlier, if the older version in not in the latest Word format, it should be converted. After acceptance of the revision, QA will again transfer the approved revision back to a generally inaccessible location in the QA directory and delete any temporary working versions in order to preserve only the exact and complete version in actual use.

## 3. Elements Of An SOP/Method

3.1 Elements

3.1.1 Almost all SOPs and methods are referred to in the general sense as SOPs. However, in some sense, they differ.

3.1.2 SOP formats are more general and free-form, not requiring the same specific elements as a Method. SOPs need only have the appropriate cover (title and revision number as on the cover of this SOP), header information, table of contents, introduction and scope, and specific operating procedures (including any appropriate appendices). Other elements may be present, depending upon the subject, but since SOPs will cover rather broad-ranging topics, no repetitive elements other than the above are currently considered necessary.

3.1.3 Methods contain the appropriate cover (title and revision number as on the cover of this SOP), header information, table of contents, introduction and scope, equipment, reagents,

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specific operating procedures, calibration and quality control (including corrective actions), and any appropriate appendices. They should also include data package assembly information and run sequences. Appendices should include preparation of standard solutions, a Method QC Table and a procedural flow chart.

3.1.4 The following elements are required by the National Environmental Laboratory Accreditation Program. Further discussion of some of these elements is presented later in this section. If not discussed directly or included within the template, it is incumbent upon the author to incorporate these elements into each relevant SOP.

1) identification of the test method;
2) applicable matrix or matrices;
3) detection limit;
4) scope and application, including components to be analyzed;
5) personnel qualifications
6) summary of the test method;
7) definitions;
8) interferences;
9) safety;
10) troubleshooting;
11) equipment and supplies including computer hardware and software (versions);
12) reagents and standards;
13) sample collection, preservation, shipment and storage;
14) quality control;
15) calibration and standardization;
16) procedure;
17) data analysis and calculations;
18) method performance;
19) pollution prevention;
20) data assessment and acceptance criteria for quality control measures;
21) corrective actions for out-of-control data;
22) contingencies for handling out-of-control or unacceptable data;
23) waste management;
24) references;
25) any tables, diagrams, flowcharts and validation data.

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3.1.5 All of the SOP/Method templates contain a title page. The title page consists of the following features:

- The laboratory name
- The SOP/Method number (assigned by QA Officer)
- The title of the SOP including EPA, SW846, Standard Methods or other method number reference when appropriate
- The revision history (revision number and date of approved revision)
- Signature of author of the most recent reviser and date signed
- Signature of managerial reviewers (minimally, the QA Officer and Lab Director but may include the Divisional Manager, Technical Director, or other key reviewer)

3.1.6 All of the SOP/Method templates contain a header record which identifies the SOP/Method number, revision, date, page number and pages, and the method or revision it replaces (if any), such as for this SOP. The header should appear on all pages except the cover page and any pages that may be attached as appendices which are not part of the document itself. This record is not readily apparent in the "normal" mode of Word templates but must be completed by the author.

3.1.6.1 In Word, choose [View][Header/Footer]. Then fill in the appropriate information, and [Close]. This information may be modified later by following the same steps. It may also be modified by using the [Page Layout] selection from the [View] mode and double-clicking the appropriate section.

3.1.7 Though not required, it is preferred that the SOP also contain a footer which identifies the laboratory. It is preferred that both header and footer are separated from the document text by a double line followed (header) or prefaced (footer) by a carriage return, such as on this SOP to separate the header or footer from the text.

3.1.8 All of the SOP/Method templates contain a Table of Contents. The table of contents will be titled as such and include the header information. It should enumerate all of the major sections of the SOP and where they are located, including appendices.

3.1.9 All SOPs/Methods contain an Introduction and Scope. This section should include a brief description of the process delineated in the rest of the text. Where the process described varies from an accepted methodology (such as SW 846 or CLP), the variations should be clearly depicted in this section.

3.1.10 In methods, sample collection. storage, and holding times should be clearly outlined.

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3.1.11 A part defining terms, particularly those which are specific to that procedure and may not be familiar to all readers is a valuable element of any procedure must be include. This section is a standard part of the templates.

3.1.12 All SOPs/Methods contain a section called Equipment List and Standards (and/or reagents). All equipment and solutions necessary to complete the process described should be outlined in this section. This must include computer hardware and software, including software version numbers.

3.1.13 All methods should contain either specific or referenced information on troubleshooting common problems.

3.1.14 All methods contain boiler plate language regarding analyst qualifications.

3.1.15 All SOPs/Methods contain a section called Safety Precautions and Waste Disposal. Any potential safety hazards should be depicted here as well as all waste disposal processes that may be entailed. If disposal involves pouring the waste into a collection container, that is all the description that is needed. The SOP then only need reference the waste disposal SOP for final disposal.

3.1.16 All SOPs/Methods involving analytical operations should contain a section discussing interferences.

3.1.17 Where appropriate (almost always in Methods), the document should contain a section on Calibration and Quality Control. This will discuss all elements related to calibration and calibration verification. It will also discuss QC samples, frequency of all calibration and QC samples, criteria for all of these samples (including how to calculate %D, % recovery, RPD or whatever other criteria that might be appropriate), and corrective actions should any of them fail to meet their respective criteria. This section will be where blanks are discussed and MDLs provided or referenced to an appendix. For most methods, a table should also be provided in one of the appendices which briefly outlines this same information. The bulk of the descriptive text, however, must appear in this section.

3.1.17.1

3.1.18 A section called Operation Procedures must be included in all SOPs/Methods which thoroughly describes the actual process. Some might consider this to be the heart of the procedure, where all analytical or other operational information is fully described in sufficient detail such that one who is reasonably familiar with the process could perform the procedure using only the SOP, with no special knowledge other than the basic principles involved and a general competence in the techniques. This section is the location for typical calculations and a description of data interpretation.

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3.1.19 A section should be contained in all methods called Reports. This should outline all analytical and QC reports and how they are presented, including control charts for many methods. This section should also include data package organization. If it is simpler to present some of this information in an appendix, the author may choose to use this approach. However, authors are encouraged to minimize the necessity of readers to reference too many sections of a procedure at one time to figure out all of the specifics of a process. In other words, it should be as easy to follow as possible and not force the reader to look in multiple sections of the SOP to find all of the information necessary for one relatively small part of the process.

3.1.20 Finally, SOPs and Methods, while not always required, will often contain Appendices. Two specific appendices common to most methods are a Quality Control Summary Table and a flow chart which depicts the basic steps involved in completing the process in the routine order and which includes the evaluation of successful completion of that process (i.e. "Is the QC in Control? If so, report the data. If not, what next?"). Where QC criteria are defined in various agency QA documents (i.e. DoD QSM, AFCEE, SW846, etc.) another common appendix is a table comparing the agency/method requirements.

3.2 Saving the Document

3.2.1 Save the document under a name that will be readily recognizable. Generally, QA will store SOPs using the SOP number LTL-XXXX with extensions .r00, .r01, etc. to denote the revision. An update should be renamed by incrementing the extension by 1 (i.e. .r01 becomes .r02, etc.). It is not necessary that the writer of the SOP use this convention but may save it as a normal WORD .doc file with a readily recognizable name. QA will then rename the SOP when it is returned to permanent storage.

3.2.2 In Word, this may be accomplished by selecting [File][Save as] and filling in the requested information.

3.2.3 All working copies of SOPs must be kept in the appropriate subdirectory of p:\sop\ so that everyone will know where to find them. Saving copies to other locations will result in uncontrolled versions of SOPs which may be either inadvertently used or lost, neither of which is acceptable. Completed and approved SOPs will be moved to a reserved area after approval.

3.2.4 Note that if you want to save the document to any other drive or directory than it was called from, you will have to specify that path. The same conventions should be used to store the document as were discussed earlier in section 2.

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3.3 After Completion of the Draft SOP

3.3.1 After the document has been written to the satisfaction of the author, it should be first passed to the department/division manager (unless that's who wrote it) for technical review. From there, the QA Officer and Lab Director will review and approve the document. QA will distribute all approved and signed documents. An unapproved (fully signed) SOP or Method document is not considered official.

3.3.2 Further details on the document tracking and control process are discussed in that SOP (LTL-1002).

## 4. References

*Quality Systems*, Ch. 5, 6/5/03, National Environmental Laboratory Accreditation Program Manual

*Department of Defense Quality Systems Manual for Environmental Laboratories*, final version 3, March 2005

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1002

Title: **Document Tracking and Control**

Revision history:

| Number | Date |
|---|---|
| 1 | 05/15/95 |
| 2 | 12/27/95 |
| 3 | 1/06/98 |
| 4 | 12/14/99 |
| 5 | 6/8/00 |
| 6 | 10/17/02 |
| 7 | 10/16/2004 |
| 8 | 12/7/2005 |

Written by: Harry Romberg Date: 12/7/05
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 12/13/05
Kathy Kreps, Laboratory Director

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## 1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to describe the system under which Laucks creates and tracks controlled documents. This insures that the latest, approved version is in use and that prior versions are kept on file but are not available for unauthorized use. It forbids the use of unapproved or expired copies of methods or procedural documents. This includes but is not limited to procedural SOPs, QA documents, and analytical methods. Other documents may be included under this system at Laucks' discretion.

1.2 Document Types

1.2.1 Laucks recognizes two types of documents.

- SOPs are considered to be administrative (such as this document or others dealing with data review or sample entry) or they may be analytical procedures (methods).
- Guidance and other miscellaneous documents may be generally broader in scope and utility than SOPs, examples being the laboratory QA Plan, Software Quality Assurance Plan or Chemical Hygiene Plan.

1.3 Scope

1.3.1 The protocol for initiating new documents is outlined, as well as the process for their approval. The tracking process is also outlined as is distribution to appropriate individuals and replacement of outdated copies with updated versions.

1.3.2 This SOP does not attempt to describe the actual creation of documents except to require that certain elements be present in order that the document may be tracked and controlled. Other SOPs (such as LTL-1001, Elements of SOP and Method Formats) describe the structure or other elements required for a specific type of document.

## 2. Operation Procedures

2.1 Initiation, Review and Updating of Documents

2.1.1 Most documents should be reviewed at least annually to ensure the procedures entailed in them are still accurate and relevant. Substantive changes may be made to a controlled document prior to that time and if so, the document revised using the procedures outlined below. Minor revisions or changes which to not substantially impact the process outlined in the document may wait for the annual review/revision process.

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2.1.2 On approximately a quarterly basis, QA issues an electronic Excel spreadsheet listing all of the SOPs that are due or overdue within about 1 month of its issuance. This spreadsheet is placed in a readily accessible location (p:\sop\) and all managers or other responsible parties informed that it has been issued. It is the manager's responsibility to review this spreadsheet and either make the document reviews/revisions or assign them to an appropriate individual.

2.1.3 If, upon review, a document is found to be current with the way the entailed procedure is actually implemented in the lab, it meets all method and program requirements, and there are not even minor changes or spelling/typographical errors that are discovered, the original document and the working photocopy (kept by QA) are stamped with a "Reviewed" stamp which are then initialed and dated. A copy of this stamp is located in Appendix D. QA will also annotate the SOP database to indicate the SOP has been reviewed.

> **Note:** Whereas substantive changes to SOPs and other critical documents should be made as soon as possible after the change, the annual review is the time to correct even the most minor errors as this contributes to the quality and professionalism of our documentation.

2.1.4 In order to track the status of documents, it is necessary to first be aware of what documents are in the process of being created, reviewed or revised. In order to do this, the Document Control & Update Form is used (see Appendix A). Prior to beginning the creation or revision of any SOP or other controlled document, this form should be filled out. It will be kept on file in the QA Department so that it will be known which documents are in the process of being written or revised, and who is the primary responsible person for creating, reviewing or revising it.

2.1.5 It is critical that when any substantial permanent change is made to a procedure that will require a change in the document, this form must be completed **immediately** and put on file in QA. These forms may also be issued when required in response to an audit or document review that points out deficiencies that require correction prior to the regularly scheduled review date. This form contains an implementation date at the bottom which is the date the change was made to the procedure. Often these changes are made before the SOP itself is modified and this date must reflect when this actually occurred. The SOP itself must still be updated but this allows for changes to be made in a timely fashion without the necessity for a complete SOP revision and release prior to actual implementation.

2.1.6 The form should be filled out by either the individual responsible for the creation or revision, their Department Supervisor, or Division Manager. Creation or revision of documents may also be assigned by the Laboratory Director, Technical Director, or QA

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Officer to specific individuals. The form, however, must be approved and kept on file by the QA Department.

2.1.7 Blanks forms are readily available in the new document templates in WORD if the user's Workgroup Templates file location is y:\template. A mock-up of this form is also in Appendix A of this SOP.

2.1.8 Copies of this form will be given to the responsible individual and the appropriate Division Manager. Originals will be kept on file in the QA Department. This insures that all responsible parties are informed of the initiation of the creation or revision process and when permanent changes are being made to procedures. This form should be filled out as soon as it is determined that the creation or revision of a document is necessary and a responsible party has been assigned.

2.1.9 Shortly after the Document Control Form is approved and distributed by the QA Department, an entry will be made in a database maintained by QA which tracks the status of that document. **All documents which have been previously approved but are currently in the process of being revised will remain in force until revisions have been completed and approved except for changes which have been previously approved and implemented on a document control form.**

2.2 Tracking and Control of Existing Documents

2.2.1 Most documents, particularly SOPs and administrative documents, will be assigned document numbers beginning with LTL. Exceptions to this are the laboratory QA Plan, Software QA Plan or Chemical Hygiene Plan already referenced. Other documents may be added to the document control system which may or may not use this numbering system only with the express permission and oversight of QA so that a mechanism for version control can be maintained. The scheme for numbering SOP documents then proceeds as follows: The most important designator is the "thousands" place. If it is unclear which "hundreds" place designator is appropriate, the one which appears to be most appropriate may be used. This SOP will not be considered to have been violated if an incorrect "thousands" or "hundreds" place designator was used but every effort will be made to use the correct designators in order to maintain a more logical organization. This organization, although preferable, is not necessary for actual control of the documents as long as each complete LTL designator is unique. Unique numbering is enforced by the SOP database.

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| | |
|---|---|
| LTL-0100 | Microbiology |
| LTL-1000 | QA / Administration |
| LTL-2000 | Health and Safety |
| LTL-3000 | Organic Extractions |
| | |
| LTL-4000 | Sample Control, |
| -4100 | Project Management |
| -4200 | Document Management and Reporting |
| | |
| LTL-5000 | Computer Systems (LIMS / MIS) |
| LTL-6000 | Miscellaneous |
| | |
| LTL-7000 | Metals Digestion |
| -7100 | ICP Analyses |
| -7200 | ICP/MS Analyses |
| -7300 | Graphite Furnace Analyses *(no longer used)* |
| -7400 | Flame Atomic Absorption Analyses *(no longer used)* |
| -7500 | Cold Vapor Atomic Absorption Analyses |
| -7600 | Gaseous Hydride Atomic Absorption Analyses *(no longer used)* |
| | |
| LTL-8000 | Gas Chromatography, Volatiles |
| -8100 | Gas Chromatography, Semivolatiles |
| -8200 | GC / Mass Spectrometry |
| -8300 | HPLC |
| -8400 | Other Organic Analyses |
| | |
| LTL-9000 | Conventional Chemistry- Titrimetric Analyses |
| -9100 | Conventional Chemistry- Spectrophotometric / Instrumental Analyses |
| -9200 | Conventional Chemistry- Gravimetric Analyses |

2.2.2 Original documents will always be given a revision number of 0. Subsequent revisions, no matter how minor the revision, will be incremented by one. Older documents may have violated this policy so revision numbering has been adjusted such that all subsequent revisions from implementation of this policy reflect this standard.

2.2.3 In addition to the numbering and revision documentation, the document must also be given a title which will uniquely identify the document content. If the document is an analytical method, the method reference should be incorporated into the title. One example of this might be "Organochlorine Pesticides and PCBs by SW 846 Method 8081A/8082."

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2.2.4 SOPs, Methods, and many other documents must have header information which clearly indicates the document number, revision, date of revision, and document replaced by revision. Page numbers and the total number of pages in the document are strongly recommended and usually required, although some discretion may be allowed by QA in special circumstances. The header may vary in format but must contain all required information similar to the following. SOPs which have been created using the template described in the Laucks SOP on the Elements of SOP and Method Formats will contain the header information as described but must be updated with every revision.

SOP No: LTL-xxxx
Revision: 1
Date: 12/7/2005
Page: x of xx
Replaces: 0

2.2.5 As a minimum, approved documents are signed by the author, QA, and the Laboratory Director. They may also be signed by other critical supervisory personnel as deemed appropriate by QA. In general, methods will either be written by these supervisory personnel and not require an additional signature, or they will be written by an individual (signed), reviewed by a supervisor (signed), and approved by QA and the Lab Director.

2.2.6 Once a document has successfully undergone review and been signed-off by the author of the document and all of the other appropriate individuals (Laboratory Director, QA Officer, and, where appropriate, Technical Director, Division Managers, etc.), it is added to the SOP database list. Only approved documents and their most currently approved revisions are noted on these lists. These lists are broken down by department and distributed to department supervisors with the distribution date indicated. New lists are distributed whenever a new document or revision is added.

2.2.7 A database is maintained by the QA Department which, as a minimum, will track the document number, Department, revision number (or New or Draft if the document is incomplete), responsible individual, title and SOP Manual distribution (if the document has been completed and approved). Also tracked are all of the approved revisions and revision dates. Copies of some of the screen forms are presented in Appendix B. The database interface is web based and is accessed through the laboratory's LIMS. Most reports and queries, however, are available through the actual underlying Access database. Both the interface and underlying database have restricted access limited to QA and MIS or other management personnel with either unrestricted access rights (necessary to maintain the LIMS) or specifically assigned access rights. QA is the only department actively involved with management of this particular database.

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2.2.8 Types of reports available from the SOP database include the following. Other reports may be added to the database as necessary and will be reflected in future revisions to this SOP but will not require modification of this SOP simply to allow their usage.

- table of contents for each SOP book, which are printed out whenever SOPs are released;
- photocopying reports, which are simply a list of SOPs released over a user (QA) designated date range that show all of the relevant books to which those SOPs have been assigned;
- reading lists, which are all SOPs assigned to an individual based on all SOPs assigned to the group to which that staff member is assigned;
- revision reading lists, which are just the latest revisions assigned to each staff member based on a user (QA) assigned date range;
- current Revision/Review status list which shows review dates for those SOPs that did NOT require revision during their most recent cycle as well as revision dates for those that did

2.2.9 Copies of the most current documents are kept on file in the QA Department and departmental specific documents are kept by the departmental supervisor in ring-binders which are available to all analysts and other appropriate staff. The SOP manuals are maintained in key locations throughout the laboratory. The SOP manuals contain only those SOPs pertinent to that area of the laboratory. The SOP manual locations are presented in Appendix C. These departmental copies are stamped with a "Controlled Document" stamp (See Appendix D) in red or black annotated with red pen. These copies, which are tracked by the QA department, will be replaced when a newer version has been completed and signed-off. The color of the Controlled Document Stamp and/or annotation, will be black on subsequent secondary copies and will not be directly tracked by the QA department as these documents are considered uncontrolled.

2.2.10 An "Uncontrolled" stamp (See Appendix D) also exists that is used on copies of SOPs that will <u>not</u> be tracked and updated. Any copies made of any SOPs must be stamped with either the "Controlled Document" or the "Uncontrolled" stamps or this policy will be considered to have been violated. Users of uncontrolled versions must be aware that the operative document could be revised at any time but that their copy will not be and thus could be outdated.

2.2.11 Copies of all of the current SOPs are also located on the laboratory Intranet under "SOPs". These documents are in Adobe Acrobat (.pdf) format and are all stamped

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"uncontrolled" because once published in this manner, controlling printed copies is extremely difficult. But, this does make all SOPs readily available to everyone in the lab.

2.2.12 It is the **Departmental Supervisor's** responsibility to ensure that their staff have copies of the most recent version of any document available to them. Keeping copies of outdated versions is inappropriate as they may be inadvertently used by uninformed individuals. When revised versions are issued, the old versions will be collected from the SOP books and usually disposed. In addition, the SOP book table of contents will be updated to reflect the revised SOP(s).

**2.2.13 It is inappropriate for any individual to be working from an unapproved copy of a method or procedure. This means that individuals must be working from original controlled documents and not copies. If an individual must consult an SOP, they must consult a controlled copy, which is readily available in a number of areas throughout the laboratory.**

**2.2.14 If during the course of training, it is absolutely necessary for the trainee to make a copy of an SOP, it must be approved by the area supervisor and that SOP MUST be stamped "uncontrolled". It must be returned to the supervisor within 2 weeks and the uncontrolled copy destroyed.**

2.2.15 **Individuals MUST NOT write or make notations in any controlled document.** Doing so makes that version different from every other version and thus, it is no longer a controlled document.

2.2.16 When documents are distributed to the departmental supervisor, copies of the reading lists, or in most cases revision reading lists, for the affected departments are also distributed. These reading lists are returned to the QA department by the individual staff members when completed.

2.2.17 Departmental supervisors will ensure that the most recent versions of all appropriate documents are made available to all affected staff members. When this occurs, three things must happen.

- Newly distributed versions are placed in the SOP manuals.
- The reading lists for the current documents are signed and dated as staff complete reading the SOPs. In addition, as staff new to a particular task (SOP) are trained, the departmental supervisor will ensure that they have read and signed the reading list for that SOP. This may require that the supervisor request a new SOP reading list for that staff member so that they can sign the SOP for newly assigned tasks.
- The departmental supervisor is responsible for ensuring that all outdated versions of SOPs are discarded or destroyed when the newer revisions are issued.

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2.2.18 Note that although any person capable of performing a documented task should be in possession of or have access to a current, officially assigned copy, the possession of a copy of any SOP or method does not imply that the individual in possession is qualified to perform the task detailed. They must still be properly trained in the techniques involved.

2.2.19 Note that versions of methods or SOPs which have been given to regulatory agencies or clients are generally uncontrolled in that they will not be updated except by specific arrangement. Controlled documents may be released to clients upon specific arrangement. However, the laboratory can only control the document and any updates up to the point they are provided to the client. It becomes the client's responsibility to then ensure that they are referencing the most recent versions in their own documentation.

2.3 Storage and Filing of Controlled Documents

2.3.1 Controlled documents will be kept by the QA Department. Master originals of the documents will be stored in a secure file and will generally not be used except to act as the reference copy and make intermediate "reproduction" copies.

2.3.2 Reproduction copies will be used to make subsequent copies for distribution to the laboratory and other authorities. These will be filed in QA but may not be stored in the same secure manner as the master copies.

2.3.3 Both master original and reproduction copies will be filed in order of their SOP number as defined previously.

2.3.4 Electronic versions of all controlled documents are also kept on file by QA in the QC directory and in the sop\methods subdirectory under their obvious subsequent subdirectory branches. These versions are stored in an area of the laboratory network which has limited access to designated individuals. These electronic copies will be given names as closely matched as possible to their document or SOP number. Original documents and revisions will be given the extension .R00 or .R01, etc. to indicate their revision number. Should multiple files be necessary to create a given document, they will be incorporated into a subdirectory with similar naming conventions.

2.3.5 Copies of these electronic versions of SOPs will be distributed to individuals who have been assigned a revision. These are placed in and are to be worked on in the projects directory (p:\ drive to most users) in the SOP subdirectory and are further divided into departments. No other copies of these controlled documents should be kept by laboratory staff in order that unapproved copies of the document do not proliferate.

2.3.6 Both physical and electronic revisions of old and archived SOPs are kept on file.

2.3.6.1 Original physical versions are filed in the 940 office. The current versions are

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kept separately from older revisions. When an SOP is retired completely (i.e. we are no longer doing that method) it is archived in a separate location in the 929 warehouse. That SOP is marked in the iLIMS database as archived.

2.3.6.2 Electronic copies of current and past revisions, including archived SOPs, are kept in an area of the laboratory network with limited access. They are marked with SOP and revision numbers but are otherwise all kept together in one location.

2.3.6.3 Very old electronic and physical SOP revisions may no longer be available if they were created long before the existing system was developed but most are available from their creation to the present version.

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**Appendix A**

**Document Control And Update Form**

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**Example only; get actual form from QA.**

**Laucks Testing Laboratories, Inc.**
**Document Control & Update Form**

_____ Generate New Document

_____ Review Existing Document

_____ Modify Existing Document

Document No: _____ Revision No: ___

Document Title: _____

_____

Assigned to: _____ Date: _____

Scheduled Completion Date: _____

This document has been reviewed and does **not** require modification at this time:

Reviewer: _____ Date: _____

The following changes have been determined to be necessary in order for the above document to comply with appropriate procedures and to reflect current laboratory practices *(use back of form and additional pages or attachments as necessary)*:

_____

_____

_____

_____

_____

_____

_____

_____

Submitted by: _____ Date: _____

QA Approval: _____ Date: _____

Implementation Date: _____

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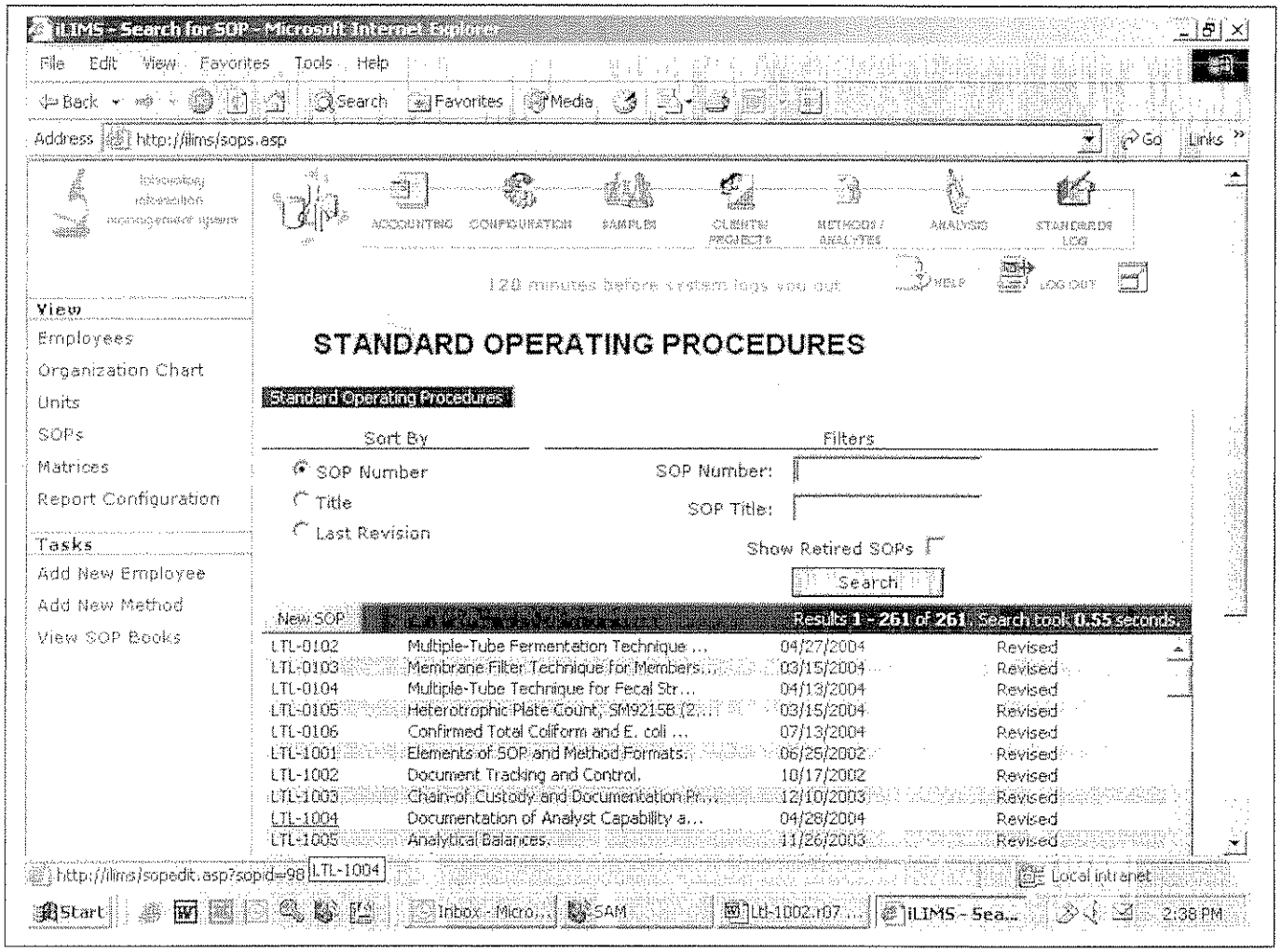
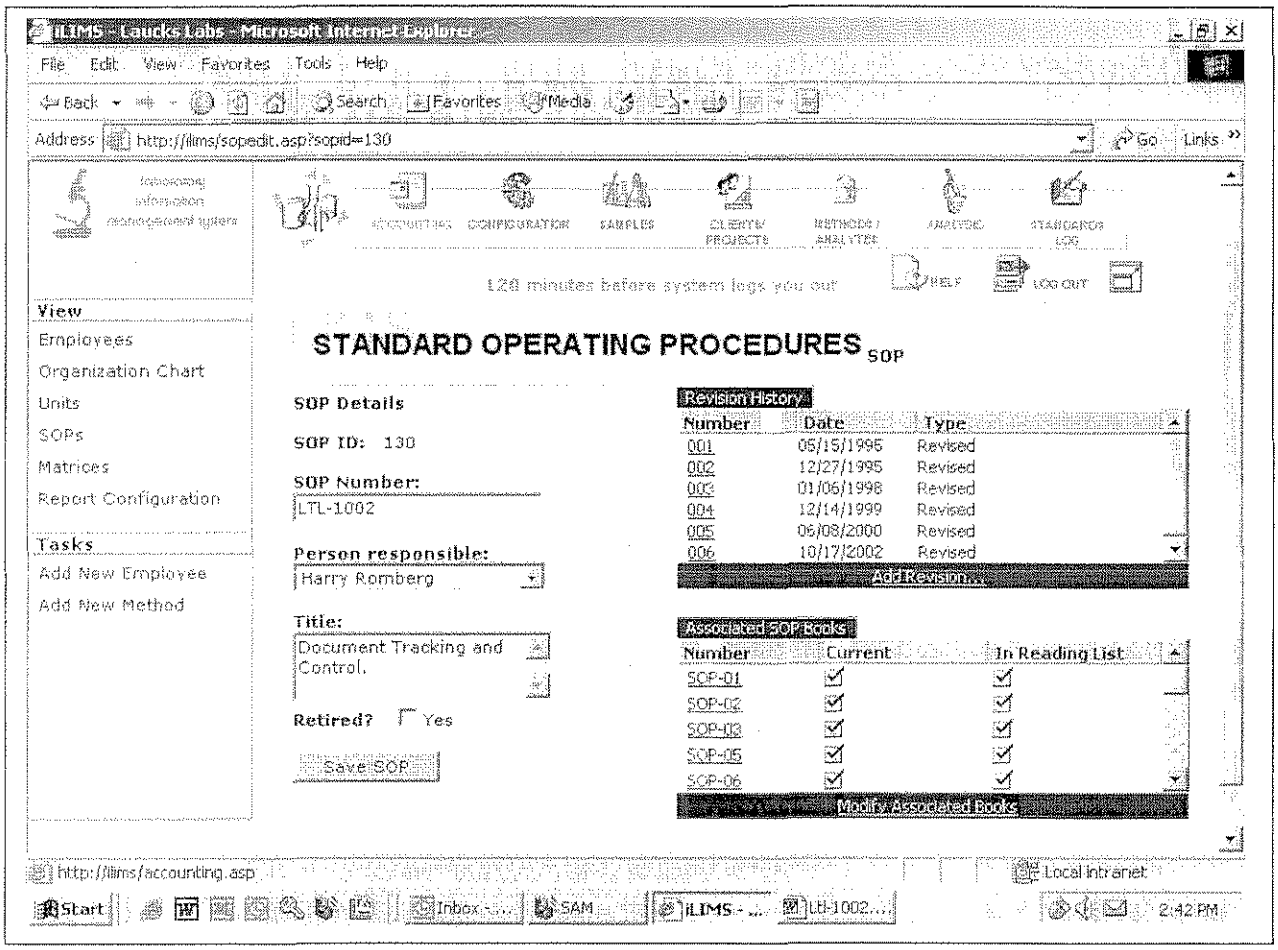
**Appendix B**

**SOP Database Screens**


iLIMS - Search for SOP - Microsoft Internet Explorer
File Edit View Favorites Tools Help
Back Search Favorites Media
Address http://ilims/sops.asp
Go Links
ACCOUNTING CONFIGURATION SAMPLES CLIENTS/ PROJECTS METHODS / ANALYTES ANALYSIS STANDARDS LOG
120 minutes before system logs you out
HELP LOG OUT
View
Employees
Organization Chart
Units
SOPs
Matrices
Report Configuration
Tasks
Add New Employee
Add New Method
View SOP Books
STANDARD OPERATING PROCEDURES
Standard Operating Procedures
Sort By
SOP Number
Title
Last Revision
Filters
SOP Number:
SOP Title:
Show Retired SOPs
Search
New SOP
Results 1 - 261 of 261. Search took 0.55 seconds.
LTL-0102 Multiple-Tube Fermentation Technique ... 04/27/2004 Revised
LTL-0103 Membrane Filter Technique for Members... 03/15/2004 Revised
LTL-0104 Multiple-Tube Technique for Fecal Str... 04/13/2004 Revised
LTL-0105 Heterotrophic Plate Count, SM9215B (2... 03/15/2004 Revised
LTL-0106 Confirmed Total Coliform and E. coli ... 07/13/2004 Revised
LTL-1001 Elements of SOP and Method Formats. 06/25/2002 Revised
LTL-1002 Document Tracking and Control. 10/17/2002 Revised
LTL-1003 Chain-of Custody and Documentation Pr... 12/10/2003 Revised
LTL-1004 Documentation of Analyst Capability a... 04/28/2004 Revised
LTL-1005 Analytical Balances. 11/26/2003 Revised
http://ilims/sopedit.asp?sopid=98 LTL-1004
Local intranet
Start
Inbox - Micro...
SAM
Ltl-1002.r07...
iLIMS - Sea...
2:38 PM


iLIMS - Laucks Labs - Microsoft Internet Explorer
File Edit View Favorites Tools Help
Back Search Favorites Media
Address http://ilims/sopedit.asp?sopid=130
Go Links
laboratory information management system
ACCOUNTING CONFIGURATION SAMPLES CLIENTS/ PROJECTS METHODS / ANALYTES ANALYTICAL STANDARDS LOG
120 minutes before system logs you out
HELP LOG OUT
View
Employees
Organization Chart
Units
SOPs
Matrices
Report Configuration
Tasks
Add New Employee
Add New Method
STANDARD OPERATING PROCEDURES SOP
SOP Details
SOP ID: 130
SOP Number:
LTL-1002
Person responsible:
Harry Romberg
Title:
Document Tracking and Control.
Retired? Yes
Save SOP
Revision History
Number Date Type
001 05/15/1995 Revised
002 12/27/1995 Revised
003 01/06/1998 Revised
004 12/14/1999 Revised
005 06/08/2000 Revised
006 10/17/2002 Revised
Add Revision...
Associated SOP Books
Number Current In Reading List
SOP-01
SOP-02
SOP-03
SOP-05
SOP-06
Modify Associated Books
http://ilims/accounting.asp
Local intranet
Start
Inbox - ...
SAM
iLIMS - ...
LTL-1002...
2:42 PM

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## Appendix C

## SOP Manual Distribution Locations

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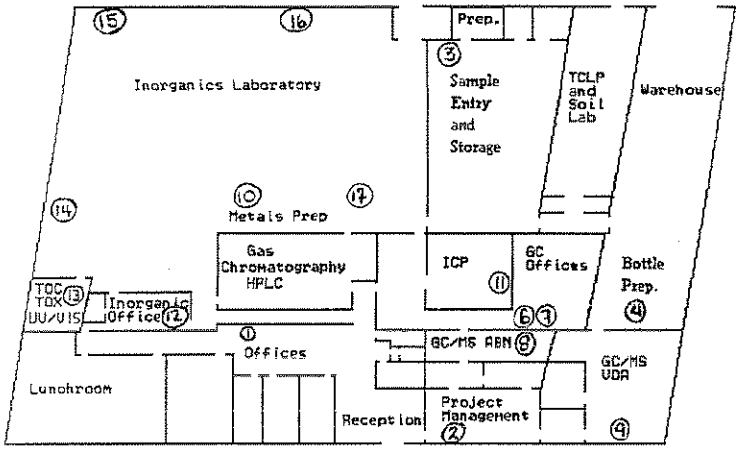
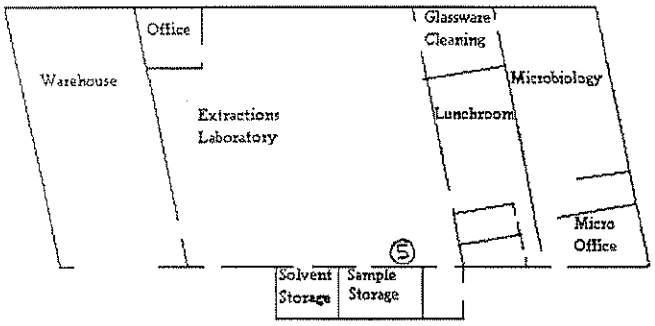
**Laucks Testing Laboratories, Inc.**
**940 South Harney St., Seattle**




**Laucks Testing Laboratories, Inc.**
**921 South Harney St., Seattle**




SOP Book Location

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## Appendix D

### Controlled Document Stamp

**Controlled Document**

**No._____ Assigned to:_____**

### Uncontrolled Stamp

**UNCONTROLLED**

### Reviewed Stamp

**Reviewed**

Does NOT require revision: ☐

Initials: _____ Date: _____

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Book: 20 Assigned to: Tetra Tech

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1003

Title: Chain-of-Custody and Documentation Procedures

Revision history:

| Number | Date |
|---|---|
| 1 | 2/13/95 |
| 2 | 2/3/98 |
| 3 | 12/10/03 |
| 4 | 6/7/2005 |
| 5 | 5/16/07 |

Written by: Harry Romberg Date: 5/16/07
Harry Romberg, Quality Assurance Officer

Reviewed by: Mike Nelson Date: 5/16/07
Mike Nelson, Technical Director

Approved by: Hugh Prentice Date: 5/18/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Description

1.1.1 This SOP is intended to describe the chain-of-custody process at Laucks, for all samples from the point of receipt until the time of disposal. It does not address actual sample receipt, entry and log-in, nor does it address any aspect of sample analysis or reporting of results except as it pertains to maintaining the chain-of-custody. The chain-of-custody process is primarily described for samples requiring secure storage and strict chain-of-custody documentation. However, most of it applies to all other samples as well.

1.1.2 The location of all samples requiring secure storage must be known at all times over the course of their possession by Laucks. Failure to maintain these conditions may result in invalidation of data on legal grounds, regardless of the technical level of data quality.

1.1.3 This process is restricted to use by, or under the supervision of analysts experienced in the process described. Each analyst or other individual requiring possession of the samples for any reason must understand the necessity of this documentation chain and be familiar with the process. Any person requiring access to the samples outside of any storage area must check them out using the described procedures.

1.1.4 Virtually all analytical staff and many others employed by Laucks are considered authorized personnel and may have access to one or more of the secure storage areas as needed for performance of their duties, at the discretion of the individual, and depending upon the nature of their duties. Removing of the samples or any aliquots thereof from the secure areas, however, requires completing the process defined for this purpose. Individuals who are not Laucks employees will not have access to samples except under the direct observation and accompaniment of staff members.

1.1.5 Laucks fully automated **L**aboratory **I**nformation **M**anagement **S**ystem, which we are calling iLIMS, includes bar-coding of sample containers, storage areas and even individuals' IDs thus enabling electronic tracking once samples have entered the lab. The physical handling of samples remains essentially the same as before bar-coding except that instead of handwritten records, electronic versions are used.

1.1.6 Additional detail on the sample entry process, including Chain-of-Custody issues, can be found in Laucks SOP LTL-4002, "Electronic Sample Entry and Log-In".

1.2 Definition of Terms

1.2.1 Custody - A sample is considered under custody if:

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- It is in the possession of an authorized person
- It is in view after being in the possession of an authorized individual
- It was in the possession of an authorized individual who then locked it up
- It is in a designated secure area which is accessible only to authorized personnel.

1.2.2 Chain of Custody - The process by which custody of a sample container is maintained and documented throughout the period that the bottle is in the possession of the laboratory. Any changes in the possession (custody) of the container must be documented in order that the chain-of-custody can be properly maintained.

## 2. Equipment List

- Any laboratory computer and label printer attached to the laboratory network with access to iLIMS
- Scanner, attached to any laboratory computer

## 3. Safety Precautions

3.1 Safety Precautions

3.1.1 No safety precautions are necessary for adherence to the items addressed by this SOP. However, in handling actual samples while operating under this document, all standards, samples and sample solutions should be handled as if they are hazardous substances.

## 4. Operation procedures

4.1 Identification of Samples Requiring Strict Chain-Of-Custody

4.1.1 Almost all samples entering the laboratory come with chain-of-custody (COC) logs, either generated by the client or by Laucks. Often these chains-of-custody are intended only for clear identification of samples and testing parameters, rather than actual custody maintenance. In fact, sample coolers often don't even have adequate custody seals and sample labels and even cooler contents sometimes don't match the COC record. Nevertheless, these custody logs must always be signed, timed and dated by the person checking the samples in and entering them into the laboratory database. These other discrepancies will be addressed later in additional sample receipt paperwork and client communications. Most of these issues are addressed elsewhere and for the purposes of this SOP, we will simply say that complete sample receipt discrepancy information will be thoroughly documented.

4.1.2 Most of the identification and tracking procedures outlined will apply to all samples. Those samples not requiring strict chain-of-custody may be stored in cold storage units or

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on shelves that are not locked-up but other than that, appropriate check in and checkout procedures remain the same. The buildings are all secure with sign-in and sign-out procedures in place for non-staff so routine security and tracking is virtually the same for all samples, even if they don't require it.

4.1.3 Full internal chain-of-custody procedures, including locked storage, will be followed for all project and other work which require such procedures. These are usually identified as CLP work or work which requires similar deliverables. These samples will usually, although not always, arrive with custody seals on the coolers and sometimes even the sample containers themselves. All DoD work under the Navy (NFESC), Army Corps of Engineers or Air Force (AFCEE) require these procedures, regardless of the type of deliverables requirements, as does any work involving pending legal action. If it is uncertain whether or not strict chain-of-custody should be maintained, these procedures should be followed.

4.2 Initiating Internal Chain-Of-Custody

4.2.1 Internal chain-of custody procedures begin when the samples are logged into the laboratory database. When the samples are logged into the system, they are stored in or near the sample entry area, in the main laboratory, in one of 5 locations. Each of these locations, as do all storage location in the laboratory, has its own bar code.

- The main walk-in cooler is for organic extractables which have not yet been transferred to the extractions laboratory and for inorganics which require refrigeration and locked storage.
- Initially, volatiles sample bottles are stored in the lockable refrigerator next to the 940 walk-in until they can be logged in and transferred to the outlying locked cold storage areas.
- The lockable GC volatiles refrigerator is located in the area between the GC room and the laboratory computer system hub.
- The lockable GC/MS volatiles refrigerator is located in the 940 warehouse area.
- The locked "cage" in the log-in area is for bottles not requiring refrigeration.

4.2.2 Additionally, samples requiring secure storage which are located in the walk-in will be on designated shelves. Those awaiting transferal to the organics extractions laboratory will be on their own designated shelf.

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4.2.3 All of these areas are secured under lock and key, the keys being in the possession of sample control and, in the case of the volatiles refrigerators, in the possession of key analysts in those areas.

4.2.4 Samples are logged into any of these areas by the sample receiving custodian. After the samples are entered into the computer, a bar-coded label is printed out and attached to the sample container.

4.2.5 Custody batches are created and used to assign bottles to specific storage locations.

4.3 Maintaining Internal Chain-Of-Custody

4.3.1 When bottles are logged out of storage areas, they will again be scanned into custody batches. Each person has their own ID so that the LIMS can associate the bottles with the individual who will maintain custody until they are returned to the appropriate storage unit. When they are returned, the analyst will use the custody batch to log the bottles back into a storage location or if the bottle contents are consumed they will be sent to a "consumed" location.

4.3.2 When bottles are removed for final disposal, they are scanned out of the storage unit, again using custody batches, and assigned a "disposed" or "return-to-client" location.

4.3.3 Soil samples which have been designated as "off-shore" or are from certain places in the mainland U.S. are required by the USDA APHIS to be treated prior to disposal. This could either mean incineration or simple heat treatment. Unless the samples are known to contain PCBs or some other hazardous material, Laucks treats them in-house by heating in a specified oven as outlined in our waste disposal SOP, LTL-2001. This oven has been given a bar-code identification just as any sample storage location. "Off-shore" soil sample containers can checked out of their storage locations through the custody batch process and checked into the oven, heat treated and then checked into a coded drum for final disposal. That coded drum can also be tracked to a specific manifest which therefore indicates the final disposition of any particular soil sample bottle.

4.3.4 Samples containing very high levels of hazardous substances, PCBs in particular, may require special treatment. On occasion, Laucks will designate specific drums for these types of samples. High PCB sample containers can be checked directly into these drums for final disposition, in this case usually incineration.

4.3.5 Thirty days after the final report has been shipped, the LIMS will automatically assign water VOA bottles to disposal.

4.3.6 Any analyst removing bottles from **any** secure storage area for the purpose of preparation or analysis or transferal to another department **must** sign (scan) the bottles out and must

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sign the bottles back in when they are returned, or must sign them into another secure storage area. Bottles must be in the possession of the analyst who signed them out at all times during this period and must not be left unattended.

4.3.7 When bottles have been signed out for final disposal the chain-of-custody process is considered to be complete.

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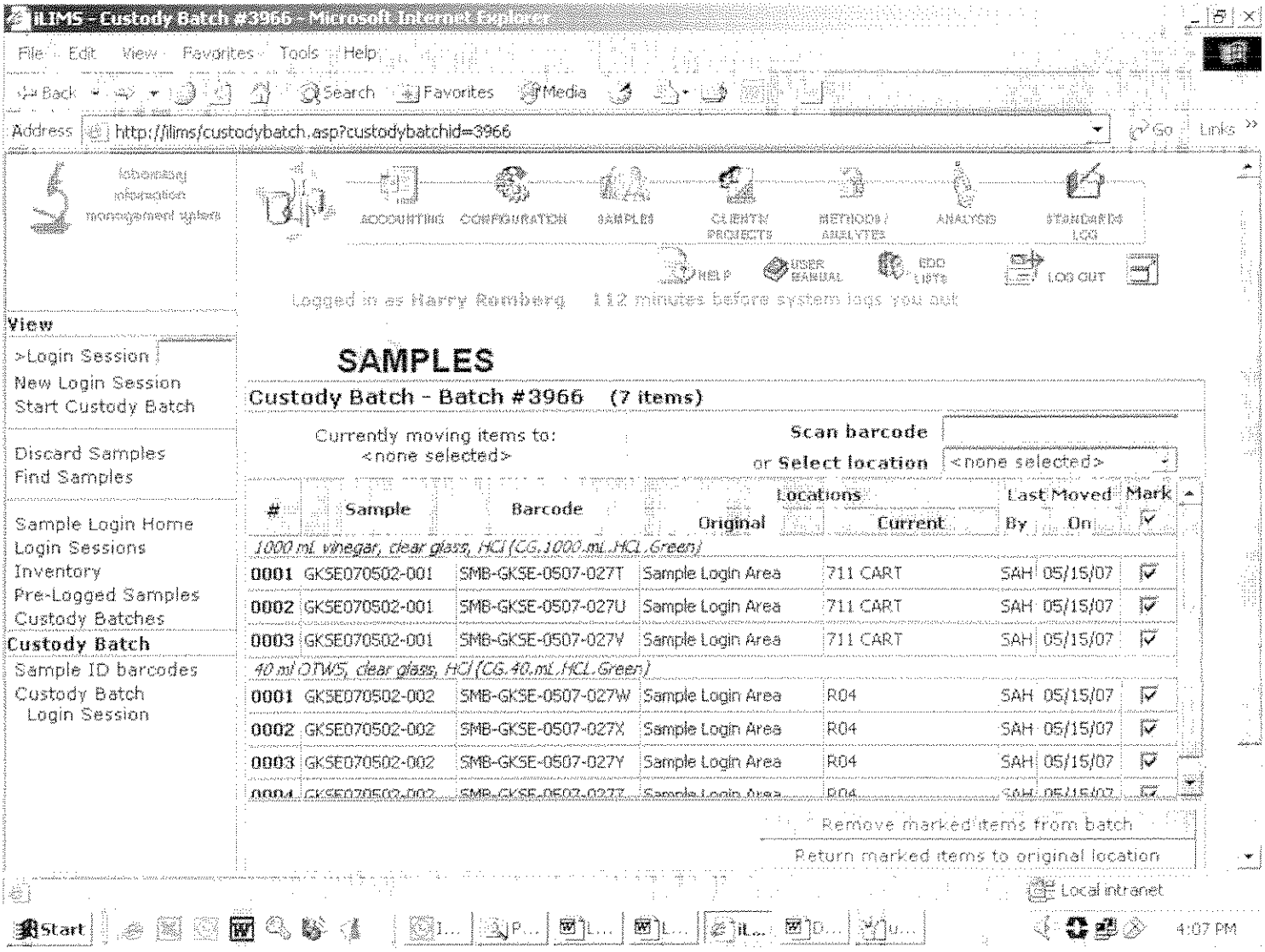
Appendix A

Custody Batch Screen



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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

Title: **Documentation of Analyst Capability and Training**

Revision history:

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Harry Romberg, Quality Assurance Officer
Date: 11/29/05

Approved by: Kathy Kreps
Kathy Kreps, Laboratory Director
Date: 12/5/05

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## 1. Introduction and Scope

1.1 Description

1.1.1 This SOP describes the way in which analyst capability is initially documented and by which the analyst is considered capable of performing independent analysis. Two practices are in place at the time of this writing.

1.1.2 This practice involves the analysis of multiple aliquots of a PE or blank spike sample and subsequent evaluation of the results. It also usually includes the completion of training checklists for the task for which the analyst is being trained. Highly experienced senior analysts with several years working at Laucks may not necessarily have the training checklists but all should have the capability demonstrations with associated NELAP compliant supervisory sign-off forms.

1.1.3 In those instances that blank spike analyses are not practical, PE sample results or other means may be substituted at the discretion of QA.

1.1.4 Continuing demonstration of proficiency usually just includes the analysis of periodic PE samples. Supervisory sign-off forms may also be used in place of (if there are no PEs) or in addition to PE samples.

1.2 Scope

1.2.1 This SOP contains discussion of initial demonstration of capability through precision and accuracy (P&A) criteria as well as PE analyses if that option is necessary. It also defines ongoing performance demonstration through the use of PE samples.

1.2.2 Both the NELAP compliant Initial Demonstration of Capability (IDOC) forms used by the Organic and Inorganic departments at Laucks are included as well as the training checklists for each department. However, whereas during periodic updates of this SOP we will attempt to incorporate any updates to these forms, they may be changed at the discretion of either QA or the department manager without necessarily resulting in an immediate SOP update.

1.2.3 Specific elements of training in safety, QA, and in each department are maintained in separate files. However, quizzes and sign-off sheets from this training are included in the respective analyst's file as demonstration that such training occurred. Specifics of these types of training are not within the scope of this SOP.

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## 2. Definitions

- **PE** or **PT** - Performance Evaluation or Performance Testing – Usually refers to sample(s) submitted either to the laboratory or individual as blind checks to assess how well the system or individual is capable of doing a particular analysis. These may be submitted as single blind samples where the tested individual knows they are check samples but does not know the true values or as double blinds where the individual does not know they are check samples.
- **IDOC** – Initial Demonstration of Capability – The demonstration that the analyst or method is capable of obtaining results within required criteria. For analyst documentation, this is accompanied with an IDOC form which formally recognizes this performance by the appropriate levels of management and QA.
- **CDOC** – Continuing Demonstration of Capability – The demonstration by the analyst that they remain capable of producing accurate results either through the analysis of PT samples, new P&A studies, or by other means acceptable to QA.
- **P&A** - Precision and Accuracy – In this context, P&A testing is generally considered to be the measurement of how reproducibly and how accurately the individual is able to perform a particular analysis on either a check sample of known or unknown levels of target analyte. This is usually done by analyzing at least 4 replicates at mid-level concentrations and observing both the recovery and relative standard deviation.
- **Trainer** - An individual who has documentation demonstrating experience recognition or successful completion of competency and has been performing the task/method for a minimum of 3 months experience for login, sample preparation, and reporting and a minimum of 6 months for analytical instrumentation operation and analysis reporting. Some methods may require additional experience before one can be considered a trainer.

## 3. Responsibilities

3.1 Analyst

3.1.1 It is the responsibility of the analyst to complete all of the items of their required training in an appropriate timeframe as required by their manager, safety and QA.

3.1.2 The analyst must complete all demonstration of capability items outlined in this SOP in a manner consistent with the analytical SOP.

3.1.3 To be considered fully trained for a particular analysis, the analyst must read all relevant SOPs.

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3.1.4 The analyst in conjunction with an assigned trainer and department manager must help to insure that all training checklists are completed to the satisfaction of their trainer and manager.

3.1.5 The analyst must periodically (at least annually) demonstrate continuing capability by analyzing a PE study for each method for which they are considered qualified, if such exists, or by some other documented demonstration if not. For the most part, the only analyses for which continuing demonstrations of capability are documented through the use of PT samples are those analyses identified in the NELAP document or in other agency requirements.

3.1.6 The analyst must regularly perform all required method QC, including matrix and blank spikes and laboratory control sample analyses, which may also be used to qualify them for demonstration of capability.

3.2 Supervisor

3.2.1 It is the supervisor's responsibility to ensure that their analysts are all initially qualified to perform an analysis including ensuring that they have analyzed all required PE samples and performed all required P&A studies for the methods for which they will be doing analyses. Parts of this responsibility can be delegated but it is the supervisor's ultimate responsibility to make sure it happens.

3.2.2 It is the supervisors responsibility to ensure that all analysts have participated in applicable QA and safety training.

3.2.3 It is the supervisor's responsibility to ensure that on a continuing basis, at least annually, that analysts who are to be considered capable of performing an analysis, have performed within limits on at least one PE study for analyses for which such are available. For the most part, the only analyses for which continuing demonstrations of capability are documented through the use of PT samples are those analyses identified in the NELAP document or in other agency requirements.

3.2.4 It is the supervisor's responsibility to ensure that other training has occurred, whether that means peer training, reading, quizzes, completed checklists, etc.

3.2.5 It is the supervisor's responsibility to develop and maintain current departmental training materials, such as checklists, quizzes, etc.

3.2.6 It is the supervisor's responsibility to ensure that the analyst's training file has been updated with the most current PE or P&A data as well as any quizzes or checklists that are considered part of their departmental training. This responsibility includes the completion of IDOC and CDOC forms. The supervisor is responsible for signing these

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forms formally designating that analyst as being qualified. Whereas the most of the supervisors duties can be delegated, the actual formal recognition and signature should not be.

3.2.7 It is the supervisors responsibility to designate a qualified individual(s) to train personnel for their new task/assignment.

3.2.8 Supervisors also maintain departmental training files which can supplement the ones maintained by QA. These may either be files based on the individual or the analysis but both must be clearly identified and retrievable.

3.3 QA

3.3.1 QA maintains training files (except for Extractions where the supervisor maintains the files due to the location of the extractions facility). Analysis specific training files are also frequently maintained by the department managers.

3.3.2 QA periodically audits training files to ensure appropriate training is being maintained.

3.3.3 QA oversees the purchasing and coordination of PT studies.

3.3.4 QA reviews PT and P&A studies to ensure criteria have been met. QA also signs the IDOC and CDOC forms when provided by the department manager or their designee.

3.3.5 QA works with managers to assist in developing training materials.

3.3.6 QA provides training to staff in QA issues and ensures that documentation of this training is in the staff training file.

3.4 Trainer

3.4.1 Completes applicable staff training documents during the training process.

3.4.2 Reviews documentation with the individual and the supervisor to ensure timely and accurate review of progress and documentation.

## 4. Operation procedures

4.1 Overview

4.1.1 New staff at Laucks undergo a safety training which includes a safety tour and explanation of Laucks safety program followed by a safety video on their first day of employment. These are documented in the QA training files.

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4.1.2 New staff also undergo basic email and computer training, usually also on their first day of employment. This is also documented in the QA training files.

4.1.3 QA conducts a QA/ethics orientation, usually after the staff member has been on staff for at least a few days and can more easily relate to the discussion. This training is also documented in the QA training files.

4.1.4 Other types of training (LIMS, first aid, etc.) occur from time-to-time and these are also documented in QA training files.

4.1.5 At least four items are generally considered necessary to assert that an analyst is properly trained for a specific job. Training checklists specific to the task, signed current SOP or SOP revision reading lists, precision and accuracy demonstrations or recent PT results (for continuing demonstrations), and signed Initial and/or Continuing Demonstration of Capability forms are all elements that most analysts should have completed in order to be fully qualified to perform a given task.

4.2 Training Checklists

4.2.1 Whether an analyst has experience from another laboratory or is a current or new staff member but is new to a particular analysis, a training checklist should be completed for each analysis that analyst is expected to perform. An experienced analyst will quickly be able to complete the checklist under the supervision of their supervisor and trainer but they still are expected to demonstrate the skills outlined. Even a highly experienced analyst new to Laucks needs to learn the specific expectations of the way an analysis is conducted at Laucks. A new or inexperienced analyst will take longer as they will need to learn new skill sets, not just specific details, in order to meet performance expectations.

4.2.2 A trainer is assigned by the area supervisor to oversee this training and to formally recognize when the analyst is capable of performing the tasks outlined. Supervision will continue to occur until the trainer, supervisor and QA are satisfied that the analyst is capable of completing all of the checklist tasks and has demonstrated capability as otherwise outlined in this SOP.

4.2.3 Training checklists for most areas of the lab are located in Appendix A. Training checklists may also be accompanied by quizzes or other documentation.

4.3 SOP Reading Lists

4.3.1 Each analyst is expected to stay current on their QA Plan and SOP reading. A new analyst, whether or not they are experienced, may have a long list of SOPs to read to fully meet training requirements, although those specific to any particular task may not be long. An analyst that has met their initial reading requirements, however, will generally only

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need to read SOPs when they have been revised. This is a much shorter list and the SOPs generally don't change a great deal but in order to be current, an analyst must complete their reading.

4.3.2 QA generates the original reading lists for new staff members or when an analyst changes departments and distributes the list generally on the first day of employment or job change. If an analyst changes jobs, however, they should be proactive in seeking out the QA Officer in order to get the latest reading list for that department. Revision reading lists are distributed to the individual department supervisors who subsequently distribute them to staff members. In either case, the staff member must sign and return the reading lists to QA when they have been completed.

4.4 Demonstrations of Capability

4.4.1 In order to be able to perform analyses independently, analysts must formally demonstrate the capability to successfully analyze samples without assistance. Up until that time, they must be closely overseen by an analyst who is qualified and who must be the one who formally signs off on all data submitted for reporting.

4.4.2 As part of their training, analysts of all experience levels must demonstrate initially and periodically thereafter that they are capable of routinely accurate analyses. This occurs in two main ways, the precision and accuracy (P&A) studies and proficiency testing (PT) sample analysis. Documentation of both initial and continuing demonstrations (P&A studies and PT samples) may be kept either in that individual's training file or in files specific to the analysis.

4.4.2.1 Precision and Accuracy (P&A) studies are generally performed on 4 aliquots of a reference material or blank spike of known concentration. The results are evaluated for both recovery and reproducibility and the analyst is expected to meet reasonable criteria, which vary with the analysis, for each. These criteria may be derived directly from the method or they may be assigned by the laboratory where other criteria do not exist. This is the most common and acceptable way for an analyst to initially demonstrate their capability to consistently and accurately analyze samples. P&A studies may also be used to demonstrate continuing capability but generally the more common practice is to use PT samples.

4.4.2.2 The analysis of PT samples may be used for initially demonstrating capability if P&A studies cannot be performed for one reason or another. They may also be used in conjunction with a P&A study as additional evidence of initial capability. PT sample analyses are most commonly used to demonstrate continuing capability, after the initial P&A study has been performed. Continuing demonstrations of capability should be performed at least annually per method for which the analyst is to be qualified. PT results should be within the vendor's statistically determined limits.

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4.4.2.3 For analyses where performing a P&A study is not practical because there is no source of standards (i.e. pH, assorted solids analyses or most other gravimetric tests, conductivity, etc.) the analyst may use any of the following to demonstrate proficiency. The analyst must not be assisted in any way for these demonstrations. The specifics of any of these demonstrations except for the default P&A study must be defined in the specific method SOP.

- replicate PT samples; these can be the same PT sample or multiple samples as long as accuracy and reproducibility are demonstrated.

- replicate sample analyses, where there is enough of the respective analyte to produce reasonable positive values (i.e. the samples must be well above the detection limit). This will usually be accompanied by the next bullet point.

- if even these are not possible, simply be closely observed by a skilled analyst who only observes but does not assist the trainee and who attests to their capability. This option should only be used if reasonable alternatives cannot be devised and with the approval of QA. If this option is exercised, the observer should complete a checklist of the operations observed and formally attest, in writing, that the analyst satisfactorily completed the tasks involved with no assistance. This may then be attached to the IDOC form along with data from the successfully completed analytical run. The supervisor may then sign the IDOC and return it to QA for final approval.

4.5 Initial and Continuing Demonstration of Capability Certification Statements

4.5.1 Both initial and continuing demonstration of capability forms are formal recognition by the analyst, supervisor and QA that the analyst is considered capable of performing a particular analysis. These forms closely mimic the forms in the NELAP manual, Quality Systems Chapter 5, Appendix C. Copies of the forms Laucks uses are located in Appendix A of this SOP. Slightly different versions of these forms may be used in different departments but the general format is similar to the examples provided. These forms are generally used in conjunction with the actual P&A and PT studies to recognize analyst capability but they should not supercede the need for the training checklists or the current reading lists as part of the overall capability demonstration.

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## 5. References

*Quality Systems*, Ch. 5, 6/5/03, National Environmental Laboratory Accreditation Program Manual

*Department of Defense Quality Systems Manual for Environmental Laboratories*, final version 3, March 2005

Laucks SOP
LTL-1011 Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits

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Appendix A

Initial and Continuing Demonstration of Capability Certification Statements

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**Laucks Testing Laboratories, Inc.**
**940 South Harney St., Seattle, WA 98108**

**Demonstration of Capability**
**Certification Statement**
**For**

## Analyst's Name

Date:

Method number(s):
Matrices:

We the undersigned CERTIFY that:

1. The analyst identified above, using the cited test method(s), 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(s) was (were) performed by the analyst identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the Demonstration of Capability are true, accurate, complete and self-explanatory (1.)

5. All raw data (including a copy of the 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.

| | |
|---|---|
| Analyst | Date |
| Manager | Date |
| Quality Assurance Officer | Date |

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**Laucks Testing Laboratories, Inc.**
**940 South Harney St., Seattle, WA 98108**

# Continuing Demonstration of Capability Certification Statement For

Date:

Method number(s):
Matrices: Water

We the undersigned CERTIFY that:

1. The analyst identified above, using the cited test method(s), 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(s) was (were) performed by the analyst identified on this certification.

3. A copy of the test method(s) and the laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the Demonstration of Capability are true, accurate, complete and self-explanatory (1.)

5. All raw data (including a copy of the 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.

| | |
|---|---|
| Analyst | Date |
| Manager | Date |
| Quality Assurance Officer | Date |

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Appendix B

Example Training Checklists
BOD, Technicon, IC, ICP, ICP/MS, Hg, Lachat, O&G, TOC, TOX, GC Fuels, GC Volatiles, GC/ECD, GC/MS Semivolatiles, GC/MS Volatiles, HPLC

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Caucke Testing Labs

# GC/MS VOA Analyst Training Verification Checklist

| Analyst Name: | | Date: | Trainer: | Analyst: | Supervisor |
|---|---|---|---|---|---|
| Documentation | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use the Instrument Run Log | | | | |
| 3 | Able to use Instrument Maintenance Log | | | | |
| Methods | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List method(s):* _____ | | | | |
| 5 | Has read and understands Methods (SW846, CLP, 500 & 600 series, DoD, USACOE, NFESC) | | | | |
| | *List method(s):* _____ | | | | |
| 6 | Has read and understands appropriate sections of GC Training Manual, QAP | | | | |
| Instrument Operation/Maintenance | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic GC/MS theory | | | | |
| 9 | Able to use GC, P&T, Autosampler & ChemStation | | | | |
| 10 | Able to change syringe, septa & injection port liner | | | | |
| 11 | Able to trim/change columns & perform leak check | | | | |
| 12 | Able to measure and set carrier, makeup and P&T gas flow | | | | |
| 13 | Able to clean source | | | | |
| Analytical Performance | | | | | |
| 14 | Able to prepare standards and evaluate acceptance criteria | | | | |
| 15 | Able to inject BFB | | | | |
| 16 | Able to tune instrument to meet method specifications | | | | |
| 17 | Able to analyze and generate acceptable calibration curve | | | | |
| 18 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 19 | Applies acceptance criteria for surrogates and spikes | | | | |
| 20 | Able to set up analytical runs (CLP and non-CLP) and acquire data | | | | |
| 21 | Able to obtain information on samples/analyses (test codes, MDLs, etc.) | | | | |
| 22 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 23 | Understands signal:noise and can discern what an acceptable peak is | | | | |
| 24 | Knows how to confirm detection of analytes (RT, spectra confirmation) | | | | |
| 25 | Knows reanalysis criteria | | | | |
| 26 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 27 | Knows correct reporting limit(s) for each method | | | | |
| 28 | Knows how to calculate concentration, spike amount & and detection limit | | | | |
| 29 | Knows corrective action & documentation for out-of-control events | | | | |
| 30 | Able to produce a data package (In-house, CLP & SW846) | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 31 | Has successfully analyzed four P&A samples | | | | |
| 32 | Has successfully analyzed two PE samples | | | | |
| 33 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the GC/MS volatile department and has demonstrated competency at the preceding tasks for the following methods listed below.

_____

Items found to be not satisfactory at the three-month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6-month probationary period.

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**Laucks Testing Labs**

**BOD Analyst Training Verification Checklist**

| **Analyst Name:** | | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Probe Maintenance Logs | | | | |
| **Methods** | | | | | |
| 3 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 4 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 5 | Knows location and use of Probe Manuals | | | | |
| 6 | Knows basic BOD theory, including possible interferences | | | | |
| 7 | Able to change probe membrane | | | | |
| 8 | Able to calibrate probe | | | | |
| 9 | Able to maintain BOD Incubator | | | | |
| 10 | Able to check results using the Winkler method | | | | |
| **Analytical Performance** | | | | | |
| 11 | Able to prepare Seed | | | | |
| 12 | Able to prepare D & G Solution | | | | |
| 13 | Able to prepare super-oxygenated water | | | | |
| 14 | Applies acceptance criteria for spikes | | | | |
| 15 | Able to set up determine dilutions based on initial DO or historical data | | | | |
| 16 | Able to analyze cBOD | | | | |
| 17 | Able to quantitate an analytical batch (QC & samples) | | | | |
| 18 | Knows reanalysis criteria | | | | |
| 19 | Knows correct reporting limits for method(s) | | | | |
| 20 | Knows corrective action & documentation for out of control QC events | | | | |
| 21 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** *(complete one or more of the following)* | | | | | |
| 22 | Has successfully analyzed four P&A samples (include results) | | | | |
| 23 | Has successfully analyzed two PE samples (include results) 1st PE: 2nd PE: | | | | |
| 24 | Has successfully analyzed three each of two types of QC samples (include results) | | | | |

This is to certify that _____ has been an analyst in the Inorganic BOD department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

Controlled Document
Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**Metals Mercury Analyst Training Verification Checklist**

| Analyst Name: | | Date: | Trainer: | Supervisor: | Analyst: |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* _____ | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* _____ | | | | |
| | | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 6 | Knows location and use of Instrument Manuals | | | | |
| 7 | Knows basic Mercury theory | | | | |
| 8 | Able to use the software to create a method | | | | |
| 9 | Able to use the software to set up the autosampler table | | | | |
| 10 | Able to change the sample introduction pump tubing | | | | |
| 11 | Able to properly dispose of wastes | | | | |
| 12 | NON-ROUTINE: *Able to clean and/or change the cell* | | | | |
| **Analytical Performance** | | | | | |
| 13 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 14 | Able to prepare reagents | | | | |
| 15 | Able to prepare and digest samples | | | | |
| 16 | Able to prepare, digest, analyze and generate acceptable calibration curve | | | | |
| 17 | Able to analyze CCVs/CCBs and apply QC acceptance criteria | | | | |
| 18 | Applies acceptance criteria for ICVs and spikes | | | | |
| 19 | Able to set up analytical runs & acquire data | | | | |
| 20 | Able to copy the analytical run file from the hard drive to the network | | | | |
| 21 | Able to quantitate an analytical batch (standards, CCVs, QC & samples), including calculating soil results | | | | |
| 22 | Knows reanalysis and redigestion criteria | | | | |
| 23 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 24 | Knows correct reporting limits for method(s) | | | | |
| 25 | Knows corrective action & documentation for out of control QC events | | | | |
| 26 | Able to fix the data file in preparation for producing data package | | | | |
| **Method Validation** *(complete one or more of the following)* | | | | | |
| 27 | Has successfully analyzed four P&A samples | | | | |
| 28 | Has successfully analyzed two PE samples | | | | |
| 29 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Metals Mercury department and has demonstrated competency at the preceeding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

Controlled Document
Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**IC Analyst Training Verification Checklist**

| **Analyst Name:** | | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic IC theory | | | | |
| 9 | Able to use the software to create a method | | | | |
| 10 | Able to use the software to set up the autosampler table | | | | |
| 11 | Able to recognize when instrument conditions are optimal | | | | |
| 12 | Able to properly change out nitrogen tank | | | | |
| 13 | Understands relay system and instrument controls | | | | |
| 14 | Able to properly prepare and degas eluent | | | | |
| 15 | Able to prime system | | | | |
| 16 | Able to convert the system between anions and perchlorate | | | | |
| 17 | NON-ROUTINE: *Able to maintain valve operation* | | | | |
| 18 | NON-ROUTINE: *Able to clean and/or change the Analytical/Guard columns* | | | | |
| 19 | NON-ROUTINE: *Able to recognize and remedy bubbles that have entered the system* | | | | |
| 20 | NON-ROUTINE: *Able to clean and/or change tubing* | | | | |
| 21 | NON-ROUTINE: *Able to change the filters, and know locations* | | | | |
| **Analytical Performance** | | | | | |
| 22 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 23 | Able to analyze and generate acceptable calibration curve | | | | |
| 24 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 25 | Applies acceptance criteria for spikes | | | | |
| 26 | Able to set up analytical runs & acquire data | | | | |
| 27 | Able to copy the analytical run files from the hard drive to the network | | | | |
| 28 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 29 | Knows reanalysis criteria | | | | |
| 30 | Able to determine dilutions based on conductivity data (Able to use the conductivity meter) | | | | |
| 31 | Knows correct reporting limits for method(s) | | | | |
| 32 | Knows corrective action & documentation for out of control QC events | | | | |
| 33 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 27 | Has successfully analyzed four P&A samples | | | | |
| 28 | Has successfully analyzed two PE samples | | | | |
| 29 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Inorganic IC department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

Controlled Document
Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**Metals ICP Analyst Training Verification Checklist**

| **Analyst Name:** | | **Start Date:** | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|---|
| **Documentation** | | | | | | |
| 1 | Able to use Standards Log | | | | | |
| 2 | Able to use Instrument Run Logs | | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | | |
| **Methods** | | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | | |
| | *List SOP(s):* | | | | | |
| 5 | Has read and understands EPA & State Methods | | | | | |
| | *List Method(s):* | | | | | |
| 6 | Has read and understands appropriate sections ICP Training Manual | | | | | |
| **Instrument Operation/Maintenance** | | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | | |
| 8 | Knows basic ICP theory | | | | | |
| 9 | Able to use the software to create a method | | | | | |
| 10 | Able to use the software to set up the autosampler table | | | | | |
| 11 | Able to perform profiling to meet instrument specifications | | | | | |
| 12 | Able to change the sample introduction pump tubing | | | | | |
| 13 | Able to properly dispose waste | | | | | |
| 14 | Able to clean and/or change the nebulizer/spray chamber | | | | | |
| 15 | Able to clean and/or change the torch | | | | | |
| 16 | NON-ROUTINE: *Able to clean and/or change the filters, and know locations* | | | | | |
| 17 | NON-ROUTINE: *Able to optimize the torch* | | | | | |
| **Analytical Performance** | | | | | | |
| 18 | Able to perform IDL/MDL/LR studies | | | | | |
| 19 | Able to adjust IECs | | | | | |
| 20 | Able to prepare standards & pass standard QC acceptance criteria | | | | | |
| 21 | Able to analyze and generate acceptable calibration and check standards (ICV/ICB/CRDL/LRCS/ICSA/ICSAB) | | | | | |
| 22 | Able to analyze CCVs/CCBs and apply QC acceptance criteria | | | | | |
| 23 | Applies acceptance criteria for %RSD, internal standards, and spikes | | | | | |
| 24 | Able to set up analytical runs & acquire data | | | | | |
| 25 | Able to copy the analytical run file from the hard drive to the network | | | | | |
| 26 | Able to quantitate an analytical batch (standards, CCVs, QC & samples), including calculating soil results | | | | | |
| 27 | Knows reanalysis and redigestion criteria | | | | | |
| 28 | Able to perform sample dilutions (obtaining linear results) | | | | | |
| 29 | Knows correct reporting limits for method(s) | | | | | |
| 30 | Knows corrective action & documentation for out of control QC events | | | | | |
| 31 | Able to fix the data file in preparation for producing data package | | | | | |
| **Method Validation** *(complete one or more of the following)* | | | | | | |
| 33 | Has successfully analyzed four P&A samples | | | | | |
| 34 | Has successfully analyzed two PE samples 1st PE: 2nd PE: | | | | | |
| 35 | Has successfully analyzed three each of two types of QC samples | | | | | |

This is to certify that _____ has been an analyst in the Metals ICP department and has demonstrated competency at the preceeding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

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Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**Metals ICP/MS Analyst Training Verification Checklist**

| **Analyst Name:** | | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List Method(s):* _____ | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* _____ | | | | |
| 6 | Has read and understands appropriate sections ICP/MS Training Manual | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic ICP/MS theory | | | | |
| 9 | Able to use the software to create a method | | | | |
| 10 | Able to use the software to set up the autosampler table | | | | |
| 11 | Able to perform tuning to meet method criteria | | | | |
| 12 | Able to change the vacuum pump oil | | | | |
| 13 | Able to change the sample introduction pump tubing | | | | |
| 14 | **NON-ROUTINE:** *Able to clean and/or change the cone* | | | | |
| 15 | **NON-ROUTINE:** *Able to clean and/or change the torch* | | | | |
| 16 | **NON-ROUTINE:** *Able to clean and/or change the filters, and know locations* | | | | |
| 17 | **NON-ROUTINE:** *Able to clean and/or change the copper seals* | | | | |
| **Analytical Performance** | | | | | |
| 18 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 19 | Able to analyze and generate acceptable calibration curve | | | | |
| 20 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 21 | Applies acceptance criteria for internal standards and spikes | | | | |
| 22 | Able to set up analytical runs & acquire data | | | | |
| 23 | Able to copy the analytical run file from the hard drive to the network | | | | |
| 24 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 25 | Knows reanalysis and redigestion criteria | | | | |
| 26 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 27 | Knows correct reporting limits for method(s) | | | | |
| 28 | Knows corrective action & documentation for out of control QC events | | | | |
| 29 | Able to fix the data file in preparation for producing data package | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 30 | Has successfully analyzed four P&A samples | | | | |
| 31 | Has successfully analyzed two PE samples | | | | |
| 32 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Metals ICP/MS department and has demonstrated competency at the preceeding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

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Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**Lachat Analyst Training Verification Checklist**

| **Analyst Name:** | | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| 6 | Knows differences between methods | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic UV continuous flow injection theory | | | | |
| 9 | Knows possible interferences and remedies | | | | |
| 10 | Able to convert instrument between different methods | | | | |
| 11 | Able to use the software to analyze QC, calibration, and individual samples | | | | |
| 12 | Able to use the software to set up the autosampler | | | | |
| 13 | Able to recognize when instrument conditions are optimal | | | | |
| 14 | Able to remedy flow problems | | | | |
| 15 | Able to properly change out helium tank | | | | |
| 16 | Able to adjust retention time window | | | | |
| 17 | Able to modify instrument methods | | | | |
| 18 | Able to transfer/process data in Excel | | | | |
| 19 | Able to properly dispose of waste | | | | |
| 20 | Able to archive/recover old data | | | | |
| 21 | NON-ROUTINE: *Able to clean and/or change the tubing* | | | | |
| 22 | NON-ROUTINE: *Able to clean the method manifold* | | | | |
| 23 | NON-ROUTINE: *Able to clean filters* | | | | |
| **Analytical Performance** | | | | | |
| 24 | Able to prepare reagents | | | | |
| 25 | Able to prepare samples (including leaching, TKN digestion, cyanide distillation, etc.) | | | | |
| 26 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 27 | Able to analyze and generate acceptable calibration | | | | |
| 28 | Able to analyze ICV and CCVs and apply QC acceptance criteria | | | | |
| 29 | Applies acceptance criteria for spikes | | | | |
| 30 | Able to set up analytical runs & acquire data | | | | |
| 31 | Knows reanalysis criteria | | | | |
| 32 | Able to determine dilutions | | | | |
| 33 | Knows correct reporting limits for method(s) | | | | |
| 34 | Knows corrective action & documentation for out of control QC events | | | | |
| 35 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 36 | Has successfully analyzed four P&A samples | | | | |
| 37 | Has successfully analyzed two PE samples | | | | |
| 38 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Inorganic department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

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Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**O&G Analyst Training Verification Checklist**

| **Analyst Name:** | | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| 6 | Knows the difference between methods: 413.1, 413.2, 418.1, 1664A, 9070 & 9071A | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic O&G theory, including possible interferences | | | | |
| 9 | Able to use the software to create a method | | | | |
| 10 | Able to use the software to set up the analytical run | | | | |
| 11 | Able to use the software to set up curve | | | | |
| 12 | Able to troubleshoot instrument problems | | | | |
| 13 | Able to use the sonicator for soil analysis | | | | |
| 14 | Able to calibrate and use the analytical balance | | | | |
| 15 | Able to properly dispose of waste | | | | |
| 16 | NON-ROUTINE: *Able to change internal dessicant* | | | | |
| 17 | NON-ROUTINE: *Able to align the laser* | | | | |
| 18 | NON-ROUTINE: *Able to maintain vacuum pump* | | | | |
| 19 | NON-ROUTINE: *Able to redistill freon* | | | | |
| **Analytical Performance** | | | | | |
| 20 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 21 | Able to analyze and generate acceptable calibration curve | | | | |
| 22 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 23 | Applies acceptance criteria for spikes | | | | |
| 24 | Able to set up analytical runs & acquire data | | | | |
| 25 | Able to make reagents | | | | |
| 26 | Able to quantitate an analytical batch (standards, CCVs, QC & samples), including calculating soil results | | | | |
| 27 | Knows reanalysis criteria | | | | |
| 28 | Able to determine dilutions and achieve linear recoveries | | | | |
| 29 | Knows correct reporting limits for method(s) | | | | |
| 30 | Knows corrective action & documentation for out of control QC events | | | | |
| 31 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** *(complete one or more of the following)* | | | | | |
| 32 | Has successfully analyzed four P&A samples (include results) | | | | |
| 33 | Has successfully analyzed two PE samples (include results) 1st PE: 2nd PE: | | | | |
| 34 | Has successfully analyzed three each of two types of QC samples (include results) | | | | |

This is to certify that _____ has been an analyst in the Inorganic Oil and Grease department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

Controlled Document
Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**TOC Analyst Training Verification Checklist**

| Analyst Name: | | Date: | Trainer: | Supervisor: | Analyst: |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic TOC theory, including possible interferences | | | | |
| 9 | Able to use software to setup the reagent blank | | | | |
| 10 | Able to use the software to create a method | | | | |
| 11 | Able to use the software to set up the autosampler table | | | | |
| 12 | Able to use the software to set up curve | | | | |
| 13 | Able to properly change out oxygen tank | | | | |
| 14 | Able to convert instrument between soil and water analyses | | | | |
| 15 | Able to use the analytical balance to prepare dilutions. (Can calibrate balances.) | | | | |
| 16 | Able to properly dispose of waste | | | | |
| 17 | NON-ROUTINE: *Able to maintain valve operation* | | | | |
| 18 | NON-ROUTINE: *Able to clean the UV bulb/glassware/scrubber* | | | | |
| 19 | NON-ROUTINE: *Able to change permeation dryer* | | | | |
| 20 | NON-ROUTINE: *Able to clean and/or change tubing* | | | | |
| 21 | NON-ROUTINE: *Able to diagnose other problems using the software* | | | | |
| **Analytical Performance** | | | | | |
| 22 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 23 | Able to analyze and generate acceptable calibration curve | | | | |
| 24 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 25 | Applies acceptance criteria for spikes | | | | |
| 26 | Able to set up analytical runs & acquire data | | | | |
| 27 | Able to copy the analytical run files from the hard drive to the network | | | | |
| 28 | Able to quantitate an analytical batch (standards, CCVs, QC & samples), including calculating soil results | | | | |
| 29 | Knows reanalysis criteria | | | | |
| 30 | Able to determine dilutions and achieve linear recoveries | | | | |
| 31 | Knows correct reporting limits for method(s) | | | | |
| 32 | Knows corrective action & documentation for out of control QC events | | | | |
| 33 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 34 | Has successfully analyzed four P&A samples (include results) | | | | |
| 35 | Has successfully analyzed two PE samples (include results) 1st PE: 2nd PE: | | | | |
| 36 | Has successfully analyzed three each of two types of QC samples (include results) | | | | |

This is to certify that _____ has been an analyst in the Inorganic TOC department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

Controlled Document
Book: 20 Assigned to: Tetra Tech

**Laucks Testing Labs**

**TOX Analyst Training Verification Checklist**

| **Analyst Name:** | | Date: | Trainer: | Supervisor: | Analyst: |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic TOX theory | | | | |
| 9 | Able to adjust adsorption module conditions for sample volume and flow rate | | | | |
| 10 | Able to use the software to analyze QC, Calibration, and individual samples | | | | |
| 11 | Able to use the software to set up the autoinjector | | | | |
| 12 | Able to recognize when instrument conditions are optimal | | | | |
| 13 | Able to properly change out oxygen and argon tanks | | | | |
| 15 | Able to check gas flow rate | | | | |
| 16 | Able to recover old data | | | | |
| 17 | Able to properly dispose of waste | | | | |
| 18 | NON-ROUTINE: *Able to clean cell* | | | | |
| 19 | NON-ROUTINE: *Able to clean and/or change the electrodes* | | | | |
| 20 | NON-ROUTINE: *Able to recognize and remedy air flow leaks* | | | | |
| 21 | NON-ROUTINE: *Able to clean and/or change tubing/septums/o-rings/combustion tube* | | | | |
| **Analytical Performance** | | | | | |
| 22 | Able to prepare reagents | | | | |
| 23 | Able to prepare samples | | | | |
| 24 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 25 | Able to analyze and generate acceptable calibration | | | | |
| 26 | Able to analyze ICV and CCVs and apply QC acceptance criteria | | | | |
| 27 | Applies acceptance criteria for spikes | | | | |
| 28 | Able to set up analytical runs & acquire data | | | | |
| 29 | Able to copy the data files from the hard drive to the network | | | | |
| 30 | Knows reanalysis criteria | | | | |
| 31 | Able to determine dilutions | | | | |
| 32 | Knows correct reporting limits for method(s) | | | | |
| 33 | Knows corrective action & documentation for out of control QC events | | | | |
| 34 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 35 | Has successfully analyzed four P&A samples | | | | |
| 36 | Has successfully analyzed two PE samples | | | | |
| 37 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Inorganic TOX department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

Controlled Document

Book: 20 Assigned to: Tetra Tech

Laucks Testing Labs

## Pesticide/Herbicide GC Semivolatile Analyst Training Verification Checklist

| Analyst Name: | | Date: | Trainer: | Analyst: | Supervisor |
|---|---|---|---|---|---|
| Documentation | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Log | | | | |
| 3 | Able to use Instrument Maintenance Log | | | | |
| Methods | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List method(s):* | | | | |
| 5 | Has read and understands Methods (SW846, CLP, 500 & 600 series, DoD, USACOE, NFESC) | | | | |
| | *List method(s):* | | | | |
| 6 | Has read and understands appropriate sections of GC Training Manual, QAP | | | | |
| Instrument Operation/Maintenance | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic GC theory | | | | |
| 9 | Able to use GC & Autosampler to set injection and temperature program | | | | |
| 10 | Able to change syringe, septa & injection port liner | | | | |
| 11 | Able to trim/change columns, install Y connector & perform leak check | | | | |
| 12 | Able to measure and set carrier and makeup gas flow | | | | |
| 13 | Able to change detectors | | | | |
| Analytical Performance | | | | | |
| 14 | Able to prepare standards and pass standard QC acceptance criteria | | | | |
| 15 | Able to analyze breakdown check and apply QC acceptance criteria | | | | |
| 16 | Able to analyze and generate acceptable calibration curve | | | | |
| 17 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 18 | Applies acceptance criteria for surrogates and spikes | | | | |
| 19 | Able to set up analytical runs (CLP and non-CLP) and acquire data | | | | |
| 20 | Able to get information on samples/analyses (test codes, MDLs, etc.) | | | | |
| 21 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 22 | Understands signal:noise and can discern what an acceptable peak is | | | | |
| 23 | Knows the importance of retention time windows | | | | |
| 24 | Able to overlay a standard chromatogram on a sample chromatogram | | | | |
| 25 | Knows reanalysis and re-extraction criteria | | | | |
| 26 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 27 | Knows correct reporting limit(s) for each method | | | | |
| 28 | Knows how to calculate concentration, spike amount & and detection limit | | | | |
| 29 | Knows corrective action & documentation for out-of-control events | | | | |
| 30 | Able to produce a data package (In-house, CLP & SW846) | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 31 | Has successfully analyzed four P&A samples | | | | |
| 32 | Has successfully analyzed two PE samples | | | | |
| 33 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the GC semivolatile department and has demonstrated competency at the preceding tasks for the following methods listed below.

_____

Items found to be not satisfactory at the three-month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6-month probationary period.

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Book: 20 Assigned to: Tetra Tech

Laucks Testing Labs

# Fuels Training Verification Checklist

| Analyst Name: | | Date: | Trainer: | Analyst: | Supervisor |
|---|---|---|---|---|---|
| Documentation | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Log | | | | |
| 3 | Able to use Instrument Maintenance Log | | | | |
| Methods | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List method(s):* _____ | | | | |
| 5 | Has read and understands Methods (SW846, NW, AK series) | | | | |
| | *List method(s):* _____ | | | | |
| 6 | Has read and understands appropriate sections of GC Training Manual, QAP | | | | |
| Instrument Operation/Maintenance | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic GC theory | | | | |
| 9 | Able to use GC & Autosampler to set injection and temperature program | | | | |
| 10 | Able to change syringe, septa & injection port liner | | | | |
| 11 | Able to trim/change columns, install connector & perform leak check | | | | |
| 12 | Able to measure and set carrier and makeup gas flow | | | | |
| 13 | Able to clean system & maintain detectors | | | | |
| Analytical Performance | | | | | |
| 14 | Able to prepare standards and pass standard QC acceptance criteria | | | | |
| 15 | Able to analyze RTM standard and set up hydrocarbon range | | | | |
| 16 | Able to analyze and generate acceptable calibration curve | | | | |
| 17 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 18 | Applies acceptance criteria for surrogates and spikes | | | | |
| 19 | Able to set up analytical runs and acquire data | | | | |
| 20 | Able to get information on samples/analyses (test codes, MDLs, etc.) | | | | |
| 21 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 22 | Understands signal:noise and can discern what an acceptable peak is | | | | |
| 23 | Knows the importance of retention time windows | | | | |
| 24 | Able to overlay a standard chromatogram on a sample chromatogram | | | | |
| 25 | Knows reanalysis and re-extraction criteria | | | | |
| 26 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 27 | Knows correct reporting limit(s) for each method | | | | |
| 28 | Knows how to calculate concentration, spike amount & and detection limit | | | | |
| 29 | Knows corrective action & documentation for out-of-control events | | | | |
| 30 | Able to produce a data package (In-house & SW846) | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 31 | Has successfully analyzed four P&A samples | | | | |
| 32 | Has successfully analyzed two PE samples | | | | |
| 33 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Fuels department and has demonstrated competency at the preceeding tasks for the following methods listed below.

_____

Items found to be not satisfactory at the three month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6-month probationary period.

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Book: 20 Assigned to: Tetra Tech

Laucks Testing Labs

## GC/MS Semivolatile Analyst Training Verification Checklist

| Analyst Name: | | Date: | Trainer: | Analyst: | Supervisor |
|---|---|---|---|---|---|
| Documentation | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Is familiar with the Instrument Log | | | | |
| 3 | Able to use Instrument Maintenance Log | | | | |
| Methods | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List method(s):* _____ | | | | |
| 5 | Has read and understands Methods (SW846, CLP, 500 & 600 series, DoD, USACOE, NFESC) | | | | |
| | *List method(s):* _____ | | | | |
| 6 | Has read and understands appropriate sections of GC Training Manual, QAP | | | | |
| Instrument Operation/Maintenance | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic GC/MS theory | | | | |
| 9 | Able to use GC, Integrator, Autosampler & ChemStation | | | | |
| 10 | Able to change syringe, septa & injection port liner | | | | |
| 11 | Able to trim/change columns & perform leak check | | | | |
| 12 | Able to measure and set carrier and makeup gas flow | | | | |
| 13 | Able to clean source | | | | |
| Analytical Performance | | | | | |
| 14 | Able to prepare standards and evaluate acceptance criteria | | | | |
| 15 | Able toinject DFTPP | | | | |
| 16 | Able to tune instrument to meet method specifications | | | | |
| 17 | Able to analyze and generate acceptable calibration curve | | | | |
| 18 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 19 | Applies acceptance criteria for surrogates and spikes | | | | |
| 20 | Able to set up analytical runs (CLP and non-CLP) and acquire data | | | | |
| 21 | Able to obtain information on samples/analyses (test codes, MDLs, etc.) | | | | |
| 22 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 23 | Understands signal:noise and can discern what an acceptable peak is | | | | |
| 24 | Knows how to confirm detection of analytes (RT, spectra confirmation) | | | | |
| 25 | Knows reanalysis and re-extraction criteria | | | | |
| 26 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 27 | Knows correct reporting limit(s) for each method | | | | |
| 28 | Knows how to calculate concentration, spike amount & and detection limit | | | | |
| 29 | Knows corrective action & documentation for out-of-control events | | | | |
| 30 | Able to produce a data package (In-house, CLP & SW846) | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 31 | Has successfully analyzed four P&A samples | | | | |
| 32 | Has successfully analyzed two PE samples | | | | |
| 33 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the GC/MS semivolatile department and has demonstrated competency at the preceding tasks for the following methods listed below.

_____

Items found to be not satisfactory at the three-month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6-month probationary period.

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Laucks Testing Labs

# GC VOA Analyst Training Verification Checklist

| Analyst Name: | | Date: | Trainer: | Analyst: | Supervisor |
|---|---|---|---|---|---|
| Documentation | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Log | | | | |
| 3 | Able to use Instrument Maintenance Log | | | | |
| Methods | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List method(s):* _____ | | | | |
| 5 | Has read and understands Methods (SW846, 500 & 600 series, DoD, USACOE, NFESC) | | | | |
| | *List method(s):* _____ | | | | |
| 6 | Has read and understands appropriate sections of GC Training Manual, QAP | | | | |
| Instrument Operation/Maintenance | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic GC theory | | | | |
| 9 | Able to use GC & Autosampler to set injection and temperature programs | | | | |
| 10 | Able to change syringe, septa & injection port liner | | | | |
| 11 | Able to trim/change columns, install connector & perform leak check | | | | |
| 12 | Able to measure and set carrier and makeup gas flow | | | | |
| 13 | Able to clean P&T and autosampler lines | | | | |
| 14 | Able to clean & maintain detectors | | | | |
| Analytical Performance | | | | | |
| 15 | Able to prepare standards and pass standard QC acceptance criteria | | | | |
| 16 | Able to analyze and generate acceptable calibration curve | | | | |
| 17 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 18 | Applies acceptance criteria for surrogates and spikes | | | | |
| 19 | Able to set up analytical runs and acquire data | | | | |
| 20 | Able to get information on samples/analyses (test codes, MDLs, etc.) | | | | |
| 21 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 22 | Understands signal:noise and can discern what an acceptable peak is | | | | |
| 23 | Knows the importance of retention time windows | | | | |
| 24 | Able to overlay a standard chromatogram on a sample chromatogram | | | | |
| 25 | Knows reanalysis and re-extraction criteria | | | | |
| 26 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 27 | Knows correct reporting limit(s) for each method | | | | |
| 28 | Knows how to calculate concentration, spike amount & and detection limit | | | | |
| 29 | Knows corrective action & documentation for out-of-control events | | | | |
| 30 | Able to produce a data package (In-house & SW846) | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 31 | Has successfully analyzed four P&A samples | | | | |
| 32 | Has successfully analyzed two PE samples | | | | |
| 33 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the GC VOA department and has demonstrated competency at the preceding tasks for the following methods listed below.

_____

Items found to be not satisfactory at the three-month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6-month probationary period.

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Caucke Testing Labs

# HPLC Analyst Training Verification Checklist

| Analyst Name: | | Date: | Trainer: | Analyst: | Supervisor |
|---|---|---|---|---|---|
| Documentation | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Log | | | | |
| 3 | Able to use Instrument Maintenance Log | | | | |
| Methods | | | | | |
| 4 | Has read and understands SOPs & QAP for all applicable methods | | | | |
| | *List method(s):* _____ | | | | |
| 5 | Has read and understands Methods (SW846, 500 & 600 series, DoD, USACOE, NFESC) | | | | |
| | *List method(s):* _____ | | | | |
| 6 | Has read and understands appropriate sections of HPLC Training Manual | | | | |
| Instrument Operation/Maintenance | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic HPLC theory | | | | |
| 9 | Able to use solvent delivery system to set mobile phase program | | | | |
| 10 | Able to use Autosampler Control Pad to set injection program | | | | |
| 11 | Able to change filters and guard column | | | | |
| 12 | Able to change columns & perform leak check | | | | |
| 13 | Able to measure and set mobile phase flows | | | | |
| 14 | Able to prime pumps | | | | |
| 15 | Able to prepare mobile phase (filter water, select correct solvent grade) | | | | |
| 16 | Able to change lamps | | | | |
| 17 | Able to locate the high pressure build-up | | | | |
| 18 | Able to change pump seal | | | | |
| 19 | Able to clean flow cell | | | | |
| Analytical Performance | | | | | |
| 20 | Able to prepare standards and pass standard QC acceptance criteria | | | | |
| 21 | Able to analyze and generate acceptable calibration curve | | | | |
| 22 | Able to analyze CCVs and apply QC acceptance criteria | | | | |
| 23 | Applies acceptance criteria for surrogates and spikes | | | | |
| 24 | Able to set up analytical runs and acquire data | | | | |
| 25 | Able to get information on samples/analyses (test codes, MDLs, etc.) | | | | |
| 26 | Able to quantitate an analytical batch (standards, CCVs, QC & samples) | | | | |
| 27 | Understands signal:noise and can discern what an acceptable peak is | | | | |
| 28 | Knows the importance of retention time windows | | | | |
| 29 | Able to overlay a standard chromatogram on a sample chromatogram | | | | |
| 30 | Knows reanalysis and re-extraction criteria | | | | |
| 31 | Able to perform sample dilutions (obtaining linear results) | | | | |
| 32 | Knows correct reporting limit(s) for each method | | | | |
| 33 | Knows how to calculate concentration, spike amount & and detection limit | | | | |
| 34 | Knows corrective action & documentation for out-of-control events | | | | |
| 35 | Able to produce a data package (In-house, CLP & SW846) | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 36 | Has successfully analyzed four P&A samples | | | | |
| 37 | Has successfully analyzed two PE samples | | | | |
| 38 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the HPLC department and has demonstrated competency at the preceding tasks for the following methods listed below.

_____

Items found to be not satisfactory at the three-month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6-month probationary period.

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**Laucks Testing Labs**

**Technicon Analyst Training Verification Checklist**

| **Analyst Name:** | | **Date:** | **Trainer:** | **Supervisor:** | **Analyst:** |
|---|---|---|---|---|---|
| **Documentation** | | | | | |
| 1 | Able to use Standards Log | | | | |
| 2 | Able to use Instrument Run Logs | | | | |
| 3 | Able to use Instrument Maintenance Logs | | | | |
| **Methods** | | | | | |
| 4 | Has read and understands SOPs for all applicable methods | | | | |
| | *List SOP(s):* | | | | |
| 5 | Has read and understands EPA & State Methods | | | | |
| | *List Method(s):* | | | | |
| 6 | Knows differences between cyanide methods | | | | |
| **Instrument Operation/Maintenance** | | | | | |
| 7 | Knows location and use of Instrument Manuals | | | | |
| 8 | Knows basic UV autoanalyzer theory | | | | |
| 9 | Able to setup the instrument for SW846 method and EPA method | | | | |
| 12 | Able to recognize when instrument conditions are optimal | | | | |
| 13 | Able to change out chart recorder paper | | | | |
| 14 | Knows method interferences and remedies | | | | |
| 15 | Able to adjust baseline | | | | |
| 16 | Able to obtain results from chart recorder | | | | |
| 17 | Able to process data in Excel | | | | |
| 18 | Able to properly dispose of waste | | | | |
| 19 | Able to archive/recover old data | | | | |
| 20 | **NON-ROUTINE:** *Able to troubleshoot instrument problems* | | | | |
| 21 | **NON-ROUTINE:** *Able to clean and/or change the tubing* | | | | |
| 22 | **NON-ROUTINE:** *Able to maintain chart recorder* | | | | |
| 23 | **NON-ROUTINE:** *Able to replace UV digest bulb* | | | | |
| **Analytical Performance** | | | | | |
| 24 | Able to prepare reagents | | | | |
| 25 | Able to prepare samples (both manually and online distilled) | | | | |
| 26 | Able to prepare standards & pass standard QC acceptance criteria | | | | |
| 27 | Able to analyze and generate acceptable calibration | | | | |
| 28 | Able to analyze ICV and CCVs and apply QC acceptance criteria | | | | |
| 29 | Applies acceptance criteria for spikes | | | | |
| 30 | Able to set up analytical runs & acquire data | | | | |
| 31 | Knows reanalysis criteria | | | | |
| 32 | Able to determine dilutions | | | | |
| 33 | Knows correct reporting limits for method(s) | | | | |
| 34 | Knows corrective action & documentation for out of control QC events | | | | |
| 35 | Able to put together an SDG package and a SAM package | | | | |
| **Method Validation** ***(complete one or more of the following)*** | | | | | |
| 36 | Has successfully analyzed four P&A samples | | | | |
| 37 | Has successfully analyzed two PE samples | | | | |
| 38 | Has successfully analyzed three each of two types of QC samples | | | | |

This is to certify that _____ has been an analyst in the Inorganic department and has demonstrated competency at the preceding tasks for the following methods (list below):

_____

***Items found to be not satisfactory at the 3 month interval should be discussed with the analyst and further training done. Not satisfactory items should be re-evaluated at the end of the 6 month probationary period.***

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1005

Title: **Analytical Balances**

Revision history:

| Number | Date |
|---|---|
| LTL-0005 R1 | 09/25/86 |
| LTL-0005 R2 | 11/3/87 |
| 3 | 06/22/96 |
| 4 | 5/15/98 |
| 5 | 12/15/99 |
| 6 | 11/26/03 |
| 7 | 10/18/06 |
| 8 | 10/24/07 |

Revised by: Harry Romberg Date: 10/24/07
Harry Romberg, QA Officer

Approved by: Hugh Prentice Date: 10/24/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 General

1.1.1 The most important piece of equipment in any analytical laboratory is the analytical balance. The degree of accuracy of the data is directly dependent on the accuracy of weight-prepared standards and samples. The balance should be one of the most cared for instruments in the lab. However this is not often the case.

1.1.2 The purpose of this SOP is to insure the proper use and calibration of all analytical balances in the laboratory. It involves the daily use of high and low level standard weight checks and a weekly calibration with a class "S". The results of these checks are logged in a balance logbook, thereby maintaining a record of the accuracy of that balance.

1.1.3 On an annual basis, usually in December, analytical balances are cleaned and general maintenance professionally performed by a qualified service technician. This process occurs automatically in conjunction with the service provider and Laucks purchasing and QA. It is the intent of this SOP to delineate internal calibration practices and not to provide additional specifics on externally provided service.

## 2. Equipment List

- Analytical Balance
- Manufacturer's Manual
- Balance Record Book
- Class "S" Weights

## 3. Safety Precautions

3.1 Safety

3.1.1 So as not to expose themselves or other analysts to potential harm and in order not to cross-contaminate samples, **it is critical that the individual analyst clean the balance and the balance area after each and every use of the balance**. In addition to the safety concern, one of the greatest sources of balance failure is the spilling of chemicals or samples into the mechanisms of the balance which are not cleaned up. This results in corrosion and other balance problems.

3.1.2 The analyst must not assume that the person using the balance before them cleaned up after themselves adequately and should check the area thoroughly before using the balance and clean up the area if necessary to maintain safety and reduce potential contamination.

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3.1.3 Weighing chemicals and samples is potentially hazardous. The analyst should take every precaution to avoid contact of any of these things with the skin, eyes, or through inhalation. In addition, the analyst should take precautions to see that nearby analysts or those using the balance afterwards are not inadvertently exposed.

## 4. Operation Procedure

4.1 Balance Setup

4.1.1 Most of the balances used at Laucks are of the electronic variety, although there are some mechanical balances. Although electronic balances tend to be somewhat more rugged than the mechanical variety, they are still subject to many of the same conditions which make the operation of all balances a critical component of their continued functioning.

4.1.2 The analytical balance is a fragile and delicate instrument, the operation of which is subject to shock, temperature and humidity changes. Mishandling and other insults also account for great loss in precision and accuracy (P & A). The following precautions should be observed in order to maintain and prolong the life of the balance.

4.1.3 Analytical balances should be mounted on a heavy, shockproof table, preferably one with a sufficiently large work surface. Although shock is less of a concern with electronic balances, they should still be treated with care. For virtually all of the balances currently used by Laucks, except for some of the less sensitive variety which have no leveling bubble, the balance level should be checked frequently and adjusted as necessary.

4.1.4 Balances should be located away from lab traffic and doors or windows where they might be subjected to drafts, sharp temperature changes and physical shock.

4.1.5 For mechanical balances, when the balance is not in use, the beam should be raised from the knife edges and in the lock (rest) position.

4.1.6 For all balances, nothing should be stored on the pan when the balance is not in use.

4.1.7 All doors to the weighing compartment should be closed.

4.1.8 Special precautions should be taken to avoid spillage of corrosive chemicals on the pan or inside the balance case. The interior should be kept scrupulously clean.

4.2 Balance and Weight Calibration

4.2.1 There are three levels of calibration; daily, weekly, and annual.

4.2.1.1 **Daily** - The daily calibration is done by the **first user** of the day. The user places a tare weight on the balance equivalent to a tare typically used on that balance, weighs **two**

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daily standards (class "S" weights **bracketing** those typical of the weights used on that balance) and records the weights in the balance record book. It is recommended that in order to cover the full weight range, these two weights span a wide range, that is, a small and a significantly larger weight above the tare. If either weight is outside the limits set for the standard, it must be brought to the attention of the area supervisor and QA.

4.2.1.2 **Weekly** - The balance will be checked with a range of class S weights each week by the laboratory balance custodian. This range should be from a very small weight to a high weight, possibly near the upper end of the balance's range. If a reading for a given weight exceeds the limits for that weight, the balance custodian will bring it to the attention of the area supervisor and QA. In instances where the tare weight is always near the same, the custodian will use that tare as a starting point for the range of test weights. For balances where a whole range of weights may be used, the custodian will use a range of weights that will cover the ENTIRE range of weights used on that balance. **Note:** one of the 940 weights is actually 150 grams and another 100mg and there are several other heavy weights that will allow the user to extend the calibration over almost the entire range of most of our balances so this should be considered when determining the test weight raange. This test should **NOT** preclude the daily check using a specific tare weight however.

4.2.1.3 **Annual** - Each balance will receive annual servicing and calibration by a qualified balance service representative.

4.2.2 The weights to be used for checking the balances are Class "S" weights or equivalent. The tare weight is not critical, except that it be accurately recorded.

4.2.2.1 The Class "S" Weights - These are the primary standards for checking the accuracy of the balance. They must be handled with care as they are calibrated and damage to the weights may result in inaccurate balance calibration. These weights must only be touched with the forceps supplied with the weights or with the clean white gloves also kept with the weights.

4.2.2.2 The class "S" weights are sent annually to a qualified weight re-certification service, currently Denver Instruments, although another qualified service is allowable. During this time other Class "S" weights will be used (if available) until the usual calibrated weights return. Two sets of certified weights are in use at Laucks, one residing at the 940 building and one at the 921 building. Normally, these weights are used for calibrating balances in their respective buildings. During the annual class "S" weight checks, one set is sent to be calibrated and the entire Seattle facility uses the other weight set. When the first set is returned, the second is sent out and the entire lab uses

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the first set until the second has been returned. This operation is coordinated annually by QA.

4.3 Responsibilities

4.3.1 The user is to ensure the following tasks are accomplished during the time he or she uses the balance:

- The balance is clean before use.
- The balance is level before use.
- The balance is clean and level after use.
- All weight has been removed and the balance lock lever has been returned to the proper position (for mechanical balances).
- In addition, all balances should be reset to zero when not in use.
- **Prior to use, the user should insure that the daily calibration check has been done**. If not, he or she must complete the task
- **After use, the user will insure the balance is clean and returned to the proper storage position.**
- The user will report any malfunction or failure of the daily check to the area supervisor.
- The user will mark and not use any balance which has failed calibration.

4.3.2 The balance custodian is the person assigned to perform the weekly calibration checks. The custodian's duties include:

- Performing the weekly calibration check
- Marking any balance which has failed the weekly check
- Informing the area supervisor of any balance which has failed the weekly calibration check.

4.3.3 The area supervisor will ensure that the following tasks are accomplished:

- Weekly and daily calibration checks are being performed. It is particularly important to ensure that if the individual assigned to perform the weekly checks (the balance custodian) is absent, that someone is trained and assigned to this duty.
- That any maintenance is performed for balances which do not meet specifications. This may include contacting others, such as QA, to actually correct the problem.

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- That any malfunctioning balance or balance which has failed calibration not be used until it is functioning properly.

4.4 Daily Calibration Check

4.4.1 The first user to use the balance each day is to perform the daily calibration check.

4.4.2 The user will insure he or she is familiar with the operation of the balance according to the manufacturer's manual.

4.4.3 The user will first insure that the balance level is correct by checking the balancing bubble and adjusting the legs of the balance as required.

4.4.4 The user checks the zero of the balance. If it is off, the user will adjust it according to the manufacturer's manual.

4.4.5 The user will place a tare weight on the balance which is typical of weights used on that balance (such as an empty beaker, a digestion vial or an empty VOA vial). The weight of the tare should be recorded, strictly for the record, and the balance zeroed on that weight, if it is a balance capable of zeroing on the tare (all electronic balances are so equipped). The weight of the tare is not a controlled value but is only used to indicate the level of the tare used.

4.4.6 A standard (Class "S") weight of a size at or slightly BELOW the LOWEST commonly used mass on that balance must then be added and the weight relative to the tare recorded under the appropriate day of the week in the calibration logbook. The user will then add a weight at or slightly ABOVE the HIGHEST mass typically weighed on that balance. He or she will also initial and date the entry (See Appendix I).

4.4.7 The daily weights, after taring, must not vary from its true value by more than the following amounts:

| Balance capable of weighing to: | must not vary by more than: |
|---|---|
| 0.1 gram | ±0.2 gram |
| 0.01 gram | ±0.02 gram |
| 0.001 gram | ±0.002 gram |
| 0.0001 gram | ±0.0005 gram |

4.4.7.1 **Example 1:** 1-3 gram samples are typically weighed into flasks with tare weights of 100 grams on a balance weighing to 0.0001 g. In order to perform the daily calibration check, a flask of about 100 grams is placed on the balance and the weight recorded. The balance is tared (set to zero) based on this weight. A 1.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This second weight

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must read within the limits of 0.9995 gm to 1.0005 gm. The 1 gram weight is removed and replaced with a 5 gram weight which is then weighed and must read within 4.9995 gm to 5.0005 gm.

4.4.7.2 **Example 2:** 30 gram samples are typically weighed into beakers with tare weights of 80 grams on a balance capable of weighing to 0.01 grams. In order to perform the daily calibration check, a beaker weighing about 80 grams is placed on the balance and the weight recorded. The balance is tared (set to zero) based on this weight. A 20 to 30. gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This weight must read within the limits of ±0.02 grams. This weight is removed and replaced with a higher weight (maybe 50 grams) and the weight recorded. It, too, must read within ±0.02 grams.

4.4.7.3 **Example 3:** A wide variety of masses (from 1 to 100 grams) are typically weighed on a particular balance which weighs to 0.001 grams and is used with a variety of tare weights. In order to perform the daily calibration check, any tare weight may be used as long as its not less than the minimum or greater than the largest vessel typically weighed. The tare is recorded and the balance is zeroed and a 1.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This weight must read within the limits of 0.998 gm to 1.002 gm. The 1 gram weight is removed and replaced with a 100 gram Class "S" weight which is then weighed and should read within 99.998 gm to 100.002 gm. At this higher weight, QA may specifically allow a bit wider range if necessary since there are more significant figures than required but since this is only an example and actual practice may involve different weights, we will not get specific here.

4.4.8 If the user cannot obtain a weight within the control limits established for the standard weight, he or she will bring it to the attention of the area supervisor and QA. Nothing requiring accurate weight should be weighed on a balance that does not meet calibration specifications. Any balance exceeding criteria must be clearly marked and not used until it can be brought into control.

4.4.9 An example logbook page is presented in Appendix I

4.5 Weekly Calibration Check

4.5.1 The balance custodian is the person responsible for performing the weekly calibration check and reporting problems to the area supervisor or QA. The custodian may be a different person in each area and it is the responsibility of the area supervisor to ensure that a capable balance custodian has been assigned to each area for which they are responsible. It is the responsibility of the custodian to insure that the weekly check is done even if they are not present, such as for vacation, etc.

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4.5.2 On the first day of the week, the balance custodian will perform a calibration check on each balance in the lab to which they are assigned. The results of these checks will be recorded in each balance calibration logbook. This check will be performed using the laboratory Class "S" weights.

4.5.3 The balance custodian will locate the Class "S" weights and insure they are clean. They will be returned to their proper location immediately upon completion of the calibration checks.

4.5.4 The balance custodian will insure the balance is clean.

4.5.5 The balance custodian checks the zero on the balance. If it is off, he or she will adjust it according to the manufacturer's manual.

4.5.6 At a **minimum**, the balance custodian will weigh 3 weights over the range for which the balance is used. Additional weights should be used if the range used is large in order to span the range typically used for that balance. If a specific weight (i.e. 100 mg or 30 grams) is the most often used on that balance, that weight should be included in the range of calibration. The results will be recorded to the left of the entries for the daily calibration check on separate lines. The custodian will also sign and **date** the entry. The date must include the month, day and year (See Appendix I).

4.5.7 Criteria for the weights on the weekly calibration check are as follows:

| | True value of weight | | | |
|---|---|---|---|---|
| ⬇Balance capable of weighing: ⬇ | <0.1000 - 1.0000 | 1.0000-9.99 | 10. - 50. | >50. |
| 0.1 gram | inappropriate | ±0.1 | ±0.2 | ±0.2 |
| 0.01 gram | ±0.02 | ±0.02 | ±0.02 | ±0.02 |
| 0.001 gram | ±0.002 | ±0.002 | ±0.002 | ±0.005 |
| 0.0001 gram | ±0.0005 | ±0.0005 | ±0.0020 | ±0.0050 |

4.5.8 If the balance custodian cannot obtain a reading within the control limits established for the standard weights, he or she will bring it to the attention of the area supervisor and QA.

4.5.9 An example logbook page is presented in Appendix I

4.6 Annual Calibration Check

4.6.1 The laboratory employs a reputable outside firm to perform annual maintenance and calibration of all of the analytical balances. The current firm is North West Instrument Services but any reputable vendor may be used if first approved by QA.

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## 5. References

*ASTM Standard Method of Testing,* TOP-LOADING, DIRECT READING LABORATORY SCALES AND BALANCES, Designation: E 898 – 82

*Department of Defense Quality Systems Manual*, Final Version 3, January 2006

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**APPENDIX I**

Sample Page from a Balance Logbook

| Week of | TV | Class "S" | | | Mon | Tues | Wed | Thurs | Fri | Sat | Su |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Oct. 16 - Oct. 22, 2006 | 2.0 | 2.00 | Tare → | | 54.17 | 54.16 | 54.17 | 54.18 | 54.15 | 54.17 | |
| | 10.0 | 10.00 | 2.0 gm → | | 2.00 | 2.00 | 2.00 | 2.00 | 2.00 | 2.01 | |
| OAR 10/16/06 | 50.0 | 50.00 | 50.0 gm → | | 50.00 | 49.99 | 50.01 | 50.00 | 50.00 | 50.00 | |
| | 100.0 | 100.01 | | | OAR 10/16/06 | OAR 10/17/06 | OAR 10/18/06 | OAR 10/19/06 | OAR 10/20/06 | OAR 10/21/06 | |
| Oct. 23 - Oct 29, 2006 | 2.0 | 2.00 | Tare → | | 54.17 | | | | | | |
| | 10.0 | 10.00 | 2.0 gm → | | 2.00 | | | | | | |
| OAR 10/23/06 | 50.0 | 50.00 | 50.0 gm → | | 50.00 | | | | | | |
| | 100.0 | 100.00 | | | OAR 10/23/06 | | | | | | |

Done once per week

Initials and date required

Room Standing

- Daily weights tare and 2 working weights
- Initials and dates required
- Every day balance is used

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1006

**Title: Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance**

Revision history:

| Number | Date |
|---|---|
| 1 | 05/01/96 |
| 2 | 3/20/98 |
| 3 | 12/21/99 |
| 4 | 3/2/04 |
| 5 | 6/13/05 |
| 6 | 4/13/07 |

Written by: Harry Romberg Date: 4/13/07
Harry Romberg, QA Officer

Approved by: Hugh Prentice Date: 4/16/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP provides a description of the identification and annual calibration of thermometers used for refrigerators, freezers, and ovens and the system used to record the calibrations and locations of the thermometers.

1.1.2 This SOP also provides a description of the routine monitoring, maintenance, and corrective actions to be performed when cold storage units or ovens fail to meet control limits.

## 2. Equipment List

2.1 Equipment

- NIST Traceable Standard Thermometer with a range of at least -20°C to at least 110°C.
- High temperature grease pen
- Erlenmeyer flask
- ethylene glycol or equivalent solution
- thermometers covering temperatures within the operating range of the cold storage unit, oven, or other equipment of interest.
- Water
- Chart Recorders or recording electronic monitoring devices

## 3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 During the calibration and data recording the analyst will be exposed to minimal safety hazards: boiling water, hot ovens, and mercury filled thermometers. It is incumbent on the analyst to exercise due care and caution while executing this SOP. The company will provide any protective equipment or clothing needed to assure employee safety.

3.2 Waste Disposal

3.2.1 No waste is generated in this operation. If mercury-filled thermometers are broken, however, the mercury must be collected and stored with other elemental mercury so that it may either be used in other laboratory operations or disposed.

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## 4. Thermometer Purchasing and Identification

4.1 Purchasing

4.1.1 Laucks currently has several Streck Laboratories, ERTCO and VWR Brand thermometers for cold storage monitoring but thermometers may be purchased from any reputable supplier.

4.1.2 Thermometers used for sample or standard cold storage should be accompanied by either an actual certificate of calibration against a NIST traceable thermometer or a certificate verifying that the thermometer was calibrated in accordance with standards traceable to the National Institute of Standards and Technology and does not vary by more than one scale division. They are immersed in a vial of ethylene glycol or equivalent solution to prevent freezing and to stabilize the temperature.

4.1.3 Thermometers used for oven temperature monitoring or for other purposes need to cover the expected range of the unit or process to be measured.

4.1.4 Chart recorders or other electronic devices which provide a historical record of temperatures over the range of concern are also used for some refrigeration units which require 7-day per week monitoring. They should come calibrated to a traceable source as well.

4.2 Identification

4.2.1 Thermometers are received with an individual serial number imprinted on the thermometer or may be identified in any way that distinctly distinguishes them from any other thermometer. This may involve the laboratory marking the thermometer to distinguish it from others if it does not have a distinct serial number. The use of a temperature resistant grease pen may be the most suitable for this purpose but any mechanism may be used as long as the thermometer is distinctly identified.

## 5. Calibration

5.1 Recalibration of the standard thermometer.

5.1.1 Per the 2003 NELAC Standard 5.5.5.2.1(b), the NIST traceable standard thermometer used in the chemistry areas is recalibrated approximately annually by sending it back to a manufacturer who has the capability to recalibrate thermometers to NIST specifications. Currently, Laucks uses the Barnstead International / ERTCO (formerly EverReady Thermometer Company) for recalibration services. This vendor will re-calibrate thermometers at approximately the same points at which the original calibration was performed and will take thermometers from any vendor, as long as a copy of the original calibration certificate is available.

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5.1.2 Note: At the present time, Laucks does not maintain NELAP accreditation for microbiological testing. Since holding times for microbiology are so short, our work in this area is essentially local and Washington State accreditation is adequate. Microbiology NIST traceable thermometers are recalibrated at the frequency required by the Washington State Department of Health, every 3 years.

5.1.3 At a minimum, copies of the certificates of recalibration will be kept in QA files.

5.2 Recalibration of Cold Storage and Room Temperature Thermometers

5.2.1 Refrigerator thermometers are calibrated upon receipt and annually thereafter, shortly after the return of the standard thermometer from its annual recalibration. When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. Thus an observer can easily see that a particular thermometer is currently calibrated.

5.2.2 Recording thermometers are rarely re-calibrated after their initial purchase. Each refrigeration unit that contains a recording thermometer also contains a conventional, calibrated, liquid-filled thermometer. We have found that the recording thermometers are difficult to read or to set at the precise temperature that might be observed from a conventional device. Therefore, the recording thermometer is primarily used to note changes over time (say, a weekend) when no one is present to observe the conventional thermometer. Temperature changes are easily observed from the recording devices so that an estimate can be made as to when discrepancies occurred. The calibrated thermometer is used, if need be, for comparison to the actual temperature.

5.2.3 Cold storage thermometers should not be calibrated with the standard thermometer if the standard thermometer has just been used at high temperatures (such as boiling water solutions). Thermal expansion of the thermometer at radically different temperatures may result in inaccuracies. After use at high temperatures, the standard thermometer should be allowed to stabilize at room temperature for at least 24 hours before it is used for cold storage calibration.

5.2.4 Refrigerator thermometers are placed in any functional refrigerator which is not frequently opened and has adequate space and in which the temperature is between +2°C and +6°C. Freezer thermometers are placed in a functional freezer where the temperature is between -10°C and -20°C. The temperature of the refrigerator or freezer is not especially important except that it must be accurately recorded and should be in the approximate range that refrigerators or freezers are generally kept. Cooler thermometers are already immersed in a small vial of liquid. If a thermometer is not already in such a vial it may be placed in the same Erlenmeyer flask as the standard thermometer noted below.

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5.2.5 At the same time the standard thermometer is also placed in the cold storage unit. The standard thermometer is placed in the cooler in an Erlenmeyer flask of water, ethylene glycol or other suitable liquid that will not freeze at the temperature of the unit.

5.2.6 Thermometers used for temperatures near room temperature may be calibrated in the BOD incubator using the same process.

5.2.7 The thermometers are allowed to equilibrate at least overnight (12 hours) and the temperatures read and recorded. Read the temperature of the standard thermometer first, then the individual thermometers. If several thermometers are being calibrated

> **Note:** Most thermometers are marked in 1°C or 2°C increments. This will require interpolation by the analyst to estimate intermediate temperatures.

concurrently, the traceable thermometer may be read several times over the course of recalibrating the other thermometers in order to accommodate minor temperature fluctuations and make sure the readings are as accurate as possible.

5.2.8 Temperatures are recorded on a blank hardcopy of an Excel spreadsheet along with the cold storage unit ID and location, the thermometer ID, and the date (See Appendix 1). The data are later transferred to the electronic version for storage and printing. The standard thermometer and the individual thermometer readings are recorded in the log and the difference is calculated and recorded to the nearest 0.1°C.

5.2.9 The differences in temperature between the standard thermometer and the individual thermometer are calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number (either a positive or negative value) to the individual thermometer reading will result in a "correct" temperature.

5.2.10 Correction factors are also recorded on the Cold Storage Temperature logs. An example of one of these forms is in Appendix B. The year at the top of this form changes annually without invalidating this SOP. They are located on each cold storage unit which is used for storage of environmental samples or standards.

5.2.11 When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. A similar color coded sticker will be attached to the original hardcopy of the annual calibration log so an observer (with the log) can easily know that the thermometer is currently calibrated. Other thermometers will be marked with tape, the calibration factor noted, and initialed and dated.

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5.3 Recalibration of Oven and Other Thermometers

5.3.1 Oven and other warm temperature thermometers should not be calibrated with the standard thermometer if the standard thermometer has just been used at low temperatures (such as refrigerator or freezer calibrations). Thermal expansion of the thermometer at radically different temperatures may result in inaccuracies. After use at low temperatures, the standard thermometer should be allowed to stabilize at room temperature for at least 24 hours before it is used for high temperature calibration.

5.3.2 For hot temperature calibration (generally used at temperatures too hot to touch), thermometers are calibrated in a continuously stirred boiling water bath. The standard and individual thermometers are inserted into a beaker of boiling water up to the immersion line. Thermometers which are kept entirely in the oven should be completely immersed in the boiling water bath. The thermometers will read a temperature slightly above 100°C if the bulbs of the thermometers are resting directly on the bottom of the beaker while the hotplate is in a heating mode. The thermometers are allowed to equilibrate for four-five minutes and the temperatures read to the nearest 1°C. Temperatures are recorded in an Excel spreadsheet along with the oven ID (if it was an oven thermometer), the thermometer ID, and the date (See appendix A).

5.3.3 The difference in temperature between the standard thermometer and the individual thermometer is calculated and recorded in the log as the "correction factor". The correction factor is calculated as the standard thermometer reading minus the individual thermometer reading so that by adding the resulting number (either a positive or negative value) to the individual thermometer reading will result in a "correct" temperature.

5.3.4 Correction factors may be written on the thermometer and/or on the unit with which that thermometer is used.

5.3.5 When a thermometer has been recalibrated, a small color coded sticker is attached. The color code will correspond to a particular yearly calibration. A similar color coded sticker will be attached to the original hardcopy of the annual calibration log so an observer (with the log) can easily know that the thermometer is currently calibrated. Other thermometers will be marked with tape, the calibration factor noted, and initialed and dated.

5.4 Recalibration of the Infrared Thermometer

5.4.1 Infrared thermometers are recalibrated at least annually or more frequently at the discretion of QA. These thermometers are also checked daily against another calibrated liquid-filled thermometer in order to ensure that there has not been significant changes due to a low battery charge or deteriorating electronic components. If a battery is changed, the thermometer is simply re-checked against the secondary

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thermometer. If disagreement between the IR thermometer and the secondary thermometer is still observed, the IR thermometer may need to be serviced or fully recalibrated against the traceable thermometer.

5.4.2 The NIST traceable thermometer is placed in a glass Erlenmeyer flask filled with water in a cold storage unit at least overnight (12 hours). This same measurement process is used for the daily calibration, although a secondary thermometer is used in place of the directly traceable thermometer. Since emissivity settings vary not only due to temperature fluctuations but the material being measured, calibrations and emissivity determinations should also be made from plastic and other containers similar to those typically measured.

5.4.3 The infrared thermometer is used to measure the temperature of the container while it contains the standard thermometer.

5.4.4 The emissivity of the IR thermometer is set at the level determined in the previous calibration (if any). It should read the same temperature as the standard thermometer. If it doesn't, the emissivity is adjusted until the standard thermometer and the IR thermometer agree as closely as possible. No emissivity adjustments should be made based solely on the comparison to the secondary liquid-filled thermometer. Such adjustments should only be made based on traceable thermometer calibrations.

5.4.5 This process should be followed for each type of container being measured and the emissivity setting that gives the most consistently accurate temperature reading recorded for each container type.

5.4.6 These emissivity settings, the calibration date and the person who performed the calibration are recorded on a label which is attached to the thermometer. Analysts subsequently using the thermometer must measure against similar containers and use the emissivity settings noted on the IR thermometer for that container type in order to get an accurate temperature measurement.

5.4.7 The appropriate information is also recorded on the Excel spreadsheet used for the other calibrations.

5.5 Daily Calibration Check of the Infrared Thermometer

5.5.1 Outside of its regular calibration against the NIST traceable thermometer, the infrared thermometer is checked daily at 4°C ± 2°C against a regular glass-column thermometer, which itself has been calibrated as previously discussed, in a glass, water-filled Erlenmeyer Flask. Other container types are not measured on a daily basis unless the reading off the Erlenmeyer flask at the correct (glass) emissivity setting fails to meet criteria.

5.5.2 The regular thermometer is stored in the 940 walk-in (W01) in water in an Erlenmeyer flask. The infrared thermometer is checked against the side of the Erlenmeyer below

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the water level. The infrared thermometer should agree with the regular thermometer (corrected to the NIST thermometer) within ±2°C or the discrepancy further investigated.

5.5.3 Most often, a discrepancy may be corrected by cleaning the surface of the Erlenmeyer and re-taking the measurement or by replacing the IR thermometer's batteries. Any action taken must be recorded on the calibration logsheet. If discrepancies persist but are consistent, it may just be necessary to fully recalibrate the infrared thermometer and adjust the approved emissivity setting. This should be coordinated with QA.

5.6 Chart Recording Thermometers

5.6.1 Existing chart recording thermometers are not so precise that accurate temperature readings should be made from them. Because they are not that accurate anyway, they are not sent out for recalibration unless they demonstrate a large disagreement with the standard thermometer. This is why we use standard liquid thermometers for regular workday monitoring.

5.6.2 However, these chart thermometers are excellent for showing when temperatures change significantly. So, if the standard liquid thermometers register a non-compliant temperature, the charts may be referenced to see when it changed.

## 6. Monitoring Responsibilities

6.1 Use Of Calibration Logs For Cold Storage Monitoring

6.1.1 At the time of annual calibration of the individual thermometers, the correction factor is written in the space provided on the form by QA. This correction factor (as noted) is calculated such that adding the value results in a temperature corrected to the standard thermometer. This correction factor may be positive or negative depending upon whether the specific individual thermometer read low or high when compared to the standard thermometer.

6.1.2 It is not the intent of this SOP to discuss how individuals whose responsibility it is to monitor cold storage units are assigned. It is the responsibility of departmental supervisors to ensure that this activity is occurring in their areas.

6.1.3 The person monitoring each cold storage unit will add the correction factor to the value read on the thermometer when recording the temperature. The corrected temperature is reported to the nearest 0.1°C. As noted previously, temperatures are estimated between thermometer marks.

6.1.4 The person monitoring each cold storage unit will also check the thermometer to make sure their are no breaks in the column.

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6.1.5 It is the responsibility of the person monitoring the particular unit to take immediate corrective action as noted in this SOP and on the monitoring form or to see that corrective action is initiated by informing a supervisor or QA. Any corrective actions (including simple adjustments of the cold storage unit thermostat) **must** be noted on the Cold Storage Temperature Log (Appendix B).

6.1.6 Certain cold-storage units which require 7-day monitoring (those containing samples) contain chart-recording thermometers which continuously track temperature of the units. Those responsible for the daily monitoring of cold storage units containing chart recording thermometers should check them immediately after a weekend or holiday to make sure there have been no exceptions over that time period. Also, when the charts are changed (usually by QA), they are checked to make sure no sporadic fluctuations occurred during that time period. Usually, the chart recorder lines are smooth and show little if any fluctuations in temperature. If they show change, QA must be notified immediately.

6.1.7 The standard thermometer cold storage monitoring forms change quarterly, when new cold storage units are put on-line, or when unforeseen circumstances occur which call for a new form. The individual charged with monitoring the cold storage unit will transfer the cold storage ID, the cold storage unit location, the thermometer ID, and the correction factor to the new form. That person will also turn in the completed log to QA for permanent storage.

6.2 Monitoring Ovens And Other Devices

6.2.1 When oven thermometers are calibrated, QA will mark the oven with the thermometer ID and the correction factor. Log sheets are generally not used for ovens. It is the responsibility of any analyst using an oven to check the oven temperature and adjust as necessary and to apply the correction factor when recording temperatures on data sheets. The analyst should record the beginning and ending temperature for any temperature-critical analyses (i.e. TS, TDS, TSS, etc.).

6.2.2 In other cases where thermometers are calibrated, the correction factor will be kept with the thermometer or written directly on the thermometer (generally with a piece of tape). Again, it is the responsibility of the analyst to apply the correction factor when recording temperatures on data sheets.

## 7. Specification Limits and Corrective Actions

7.1 Thermometer Criteria

7.1.1 Thermometers should not vary by more than ±5°C from the standard thermometer reading, even though a correction factor is applied. This criterion does not apply to the infrared thermometer.

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7.1.2 There should be no observable breaks in the column of any thermometer at any time during calibration or routine use.

7.2 Thermometer Corrective Actions

7.2.1 Thermometers with a break in the column must be immediately removed from use and either repaired or replaced.

7.2.2 Thermometers which read more than ±5°C from the standard thermometer reading must not be used. If they cannot be repaired or (if new) returned to the vendor, they should be disposed or clearly marked and only used for non-critical tasks. They should not be used for the storage or analysis of environmental samples or others where temperature is a critical factor.

7.3 Cold Storage Criteria

7.3.1 Refrigeration units should be in the temperature range of 4°C ± 2°C. All freezers must be <-10°C but >-20°C.

7.4 Cold Storage Corrective Actions

7.4.1 See Appendix B for an example Cold Storage Temperature Log. This log also contains the appropriate corrective actions in an abbreviated form.

7.4.1.1 If at all possible, actions should be taken **before** a unit exceeds temperature criteria so that samples or standards are not inadvertently stored outside the required limits for any significant length of time. This alleviates the far more onerous tasks of re-preparing standards or contacting clients for samples stored out-of-specifications.

7.4.2 Adjust the thermostat of the cold storage unit if necessary.

7.4.3 Defrost the cold storage unit if necessary. This may be done prior to adjusting the thermostat if there is severe icing of the unit and it is obvious that this is the cause of the temperature deviation.

7.4.4 If the above fail to correct the problem, contact the laboratory maintenance personnel, the departmental supervisor or QA to arrange for repair.

7.4.5 If it is determined that professional servicing is required this may be arranged upon direction of one of these individuals or another senior supervisor. If professional maintenance does not correct the problem, the unit may need to be replaced, again at management discretion.

7.4.6 Samples must not be stored at inappropriate temperatures. If the problem is not quickly solved, samples or standards must be transferred to another cold storage unit. Dry ice might also be purchased and placed in the cooler in sufficient quantity to adequately cool the samples. If it is determined that samples were stored at inappropriate temperatures for an extended period, it may be necessary to contact

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clients to determine the course of action they would like us to take regarding their analyses. This should be coordinated with QA, the Lab Director and project management. Standards which have been inappropriately stored will generally require disposal, generally at the discretion of QA and/or department managers.

7.4.7 Any corrective actions (including simple adjustments) **must** be noted on the Cold Storage Temperature Log (Appendix B).

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# Appendix I

## Example Calibration Spreadsheet

| Cold Storage ID | Location | Serial No. | Cal. Date | | FC-53631 NIST reading | Thermom. Reading | Correction Factor |
|---|---|---|---|---|---|---|---|
| C01 | Inorganics | ERTCO 4156 | 11/21/2006 | | 3.2 | 3.6 | -0.4 |
| C03 | extractions | B B01679 | 11/21/2006 | | 3.2 | 3.3 | -0.1 |
| C04 | GC VOA | B B03987 | 11/21/2006 | | 3.2 | 3.7 | -0.5 |
| C05 | Inorganics | B02580 | 11/21/2006 | | 3.2 | 3.1 | 0.1 |
| C06 | 940 Warehouse | B B04765 | 11/21/2006 | | 3.1 | 3.1 | 0.0 |
| C07 | 940 warehouse | VWR 172101 | 11/21/2006 | | 3.2 | 2.6 | 0.6 |
| | | | | | | | |
| R01 | Sample Entry | B B01342 | 11/21/2006 | | 3.1 | 3.1 | 0.0 |
| R02 | GC Semi Stds. | B B03534 | 11/21/2006 | | 3.2 | 3.2 | 0.0 |
| R03 | 940 Warehouse | ERTCO 2920 | 11/21/2006 | | 3.2 | 3.5 | -0.3 |
| R04 | GC/MS whse. | B B00906 | 11/21/2006 | | 3.2 | 3.2 | 0.0 |
| R05 | 940 Warehouse | B B01919 | 11/21/2006 | | 3.2 | 3.7 | -0.5 |
| R06 | extractions | B B01708 | 11/21/2006 | | 3.2 | 3.2 | 0.0 |
| R07 | extractions | B B04059 | 11/21/2006 | | 3.2 | 3.5 | -0.3 |
| R08 | Inorganics | B 03021 | 11/21/2006 | | 3.2 | 3.0 | 0.2 |
| | | | | | | | |
| W01 | 940 Walk-in | B B03928 | 11/21/2006 | | 3.1 | 3.1 | 0.0 |
| W02 | 921 Walk-in | unmarked Temp Chex | 11/21/2006 | | 3.2 | 3.6 | -0.4 |
| | | | | | | | |
| F01 | GC/VOA | B B B08543 | 11/22/2006 | | -19.2 | -19.0 | -0.2 |
| F03 | GC semivolatiles | B B B08959 | 11/22/2006 | | -19.2 | -18.5 | -0.7 |
| F05 | GC/MS VOA | Ertco 1152 | 11/22/2006 | | -19.2 | -18.2 | -1.0 |
| F06 | GC semivolatiles | Ertco 5169 | 11/22/2006 | | -19.2 | -18.5 | -0.7 |
| F07 | GC/VOA stds. | B B B08765 | 11/22/2006 | | -19.2 | -18.8 | -0.4 |
| F08 | Sample Entry | Ertco 5236 | 11/22/2006 | | -19.2 | -21.4 | 2.2 |
| F09 | GC Instrument Room | Ertco 5034 | 11/22/2006 | | -19.2 | -20.2 | 1.0 |
| | 940 warehouse | Ertco 4366 | 11/22/2006 | | -19.2 | -22.0 | 2.8 |
| | spare | | | | | | |
| | spare | Ertco 1838 | 11/22/2006 | | -19.2 | -22.1 | 2.9 |
| | spare | VWR H21301 | 11/22/2006 | | -19.2 | -21.2 | 2.0 |
| | spare | Ertco 1535 | 11/22/2006 | | -19.2 | -20.6 | 1.4 |
| | spare | B B B09215 | 11/22/2006 | | -19.2 | -21.1 | 1.9 |
| | spare | Ertco 2034 | 11/22/2006 | | -19.2 | -20.0 | 0.8 |
| | | | | | | | |
| Hg thermometer | Sample entry | BCR 2 | 11/21/2006 | | 3.1 | 3.1 | 0.0 |
| IR Thermometer (ITT-330) | | Horiba 226099 | 11/21/2006 | **Glass** | 3.5 | 3.5 | **(at E= 90 )** |

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| Cold Storage ID | Location | Serial No. | Cal. Date | | FC-53631 NIST reading | Thermom. Reading | Correction Factor | |
|---|---|---|---|---|---|---|---|---|
| | | | 11/21/2006 | Plastic | 3.5 | 3.5 | (at E= 87 ) | |
| IR Thermometer (ITT-330) | | Horiba 132013 | 11/21/2006 | Glass | 3.5 | 3.5 | (at E= 95 ) | |
| | | | 11/21/2006 | Plastic | 3.5 | 3.5 | (at E= 100 ) | |
| | | ERTCO 5124 | 11/21/2006 | | 3.2 | 3.8 | -0.6 | |
| | | ERTCO H93-203 | 11/21/2006 | | 3.2 | 4.1 | -0.9 | |
| | | no brand, long, Hg, R001 | 11/21/2006 | | 3.5 | 3.8 | -0.3 | |
| | | ERTCO H93-102 | 11/21/2006 | | 3.2 | 4.5 | -1.3 | |

| Oven/Water-bath/Other ID | Location | Serial No. | Cal. Date | | FC-53631 NIST reading | Thermom. Reading | Correction Factor | | |
|---|---|---|---|---|---|---|---|---|---|
| VWR 1320 | Extractions | 8 | | | | | 0 | | |
| VWR 1310 | | 3 | 9/15/2006 | | 97.0 | 96.0 | 1 | | |
| VWR 1330F | #4 Univ. Enterprises | L12-004 | 9/15/2006 | | 96.9 | 96.4 | 1 | | |
| VWR 1370F | | F42653 | | | | | 0 | | |
| Thelco | | Made in UK #16 | 9/15/2006 | | 97.3 | 100.8 | -4 | Deg F | |
| VWR 1300 U (TOC room) | | VWR 61066-046 | | | | | 0 | | |
| Pensky Martin (AST | VWR 61091-001 | IY7422 | | | | | 32 | Deg F | |
| VWR 1305U | | National Appliance #7 | | | | | 0 | | |
| VWR 1300 U (Extractions) | | F22592 | | | | | 0 | | |
| Blue M (Extractions) | | VWR 61066-104 | | | | | 0 | | Broken |
| Blue M (Extractions) | | F22716 | | | | | 0 | | |
| front waterbath | | Enviro-Safe 6 | | | | | | | |
| Napco Oven | | SPER Sci. | 9/15/2006 | | 97.8 | 97.5 | 0 | | |
| Digestion Area | VWR 61014-949 | 9 | | | | | 0 | | |
| VWR ASTM 9F | | IY2085 | | | | | 32 | Deg F | |
| ERDCO | Setaflash | 1SF5531/85A038 | | | | | 32 | Deg F | |
| ERDCO | Setaflash | 1SFA5531/151831 | | | | | 32 | Deg F | |

| Oven/Water-bath/Other ID | Location | Serial No. | Cal. Date | | FC-53631 NIST reading | Thermom. Reading | Correction Factor | |
|---|---|---|---|---|---|---|---|---|
| metals digestion | | 00-01 | | broken ????? | | | 0.0 | |
| BOD thermometer | BOD Incubator | BCR 1 | | | | | 0.0 | |
| | | | | | | | | |
| spare | | F22731 | | | | | 0 | |
| | | | | | | | 0.0 | |

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| Cold Storage ID | Location | Serial No. | Cal. Date | FC-53631 NIST reading | Thermom. Reading | Correction Factor | |
|---|---|---|---|---|---|---|---|
| VWR ASTM 9F | | 09107 | | | | 32.0 | Deg F |
| | | | | | | | |
| Hg mod block | | | | | | 0 | |
| | VWR 61222-504 | 26 | | | | 0 | |
| | VWR 61222-504 | 29 | | | | 0 | |
| | VWR | A42338 | 9/15/2006 | 97.4 | 98.8 | -1 | |
| | VWR | A42507 | 9/15/2006 | 97.8 | 100 | -2 | |
| | VWR | A42219 | 9/15/2006 | 97.8 | 101 | -3 | |
| | VWR | A42379 | 9/15/2006 | 97.3 | 99.3 | -2 | |
| | PG ERTCO | 6638 | 9/15/2006 | 97.9 | 99.8 | -2 | |
| | Extractions oven | T6920 | | | | 0 | |
| | | | | | | | |
| | | VWR 02429???? | | | | | |

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# Appendix II

## Example Cold Storage Temperature Log

# Cold Storage Temperature Log

**Laucks Testing Laboratories, Inc.**

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **2007** Correction Factor (*add this number when* ***recording*** *the thermometer reading):*_____°C

| Day | Month: Time | Temp. | Initials | Actions | Month: Time | Temp. | Initials | Actions | Month: Time | Temp. | Initials | Actions |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 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 | | | | | | | | | | | | |
| 26 | | | | | | | | | | | | |
| 27 | | | | | | | | | | | | |
| 28 | | | | | | | | | | | | |
| 29 | | | | | | | | | | | | |
| 30 | | | | | | | | | | | | |
| 31 | | | | | | | | | | | | |

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.

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Corrective action includes
1) Adjust the temperature of the thermostat
2) Defrost the refrigerator or freezer
3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer
4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of.

**Control Limits**

| | |
|---|---|
| **Refrigerators** | **4°C ± 2°C** |
| **Freezers** | **-10°C to -20°C** |
| **BOD Incubator** | **20°C ± 1°C** |

Any and all actions **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of the page with the date the action occurred.

Samples **MUST NOT** be stored in units which are not maintaining the proper temperature.

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# Appendix III

## Example Infrared Thermometer Daily Check Log

# Infrared Thermometer Daily Check Log

**Laucks Testing Laboratories, Inc.**

Location: Sample Receiving  Checked against standard glass thermometer: BCR 2

Year: **2007**  Thermometer ID: **A** Horiba 226099  Emissivity Setting:

Thermometer ID: **B** Horiba 132013  Emissivity Setting:

| | Month: | | | | | Month: | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| **Day** | **Time** | **Glass Thermometer Temp. (°C)** | **IR Thermometer Temp. (°C)** | **Initials** | **Actions** | **Time** | **Glass Thermometer Temp. (°C)** | **IR Thermometer Temp. (°C)** | **Initials** | **Actions** |
| 1 | | | | | | | | | | |
| 2 | | | | | | | | | | |
| 3 | | | | | | | | | | |
| 4 | | | | | | | | | | |
| 5 | | | | | | | | | | |
| 6 | | | | | | | | | | |
| 7 | | | | | | | | | | |
| 8 | | | | | | | | | | |
| 9 | | | | | | | | | | |
| 10 | | | | | | | | | | |
| 11 | | | | | | | | | | |
| 12 | | | | | | | | | | |
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| 14 | | | | | | | | | | |
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| 18 | | | | | | | | | | |
| 19 | | | | | | | | | | |
| 20 | | | | | | | | | | |
| 21 | | | | | | | | | | |
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| 26 | | | | | | | | | | |
| 27 | | | | | | | | | | |
| 28 | | | | | | | | | | |
| 29 | | | | | | | | | | |
| 30 | | | | | | | | | | |
| 31 | | | | | | | | | | |

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.  Q:\TEMPLATS\LTLFORMS\IR_CHK1.DOC  12/29/2003

If IR thermometer exceeds ±2°C of the standard glass thermometer, corrective action must be taken. Corrective action includes

1) Change batteries  2) Clean erlenmeyer surface and read again  3) Contact the appropriate laboratory maintenance personnel, the departmental supervisor, and/or the QA Officer

4) If the IR thermometer is not working properly, the glass thermometer **must** be used.

Any and all actions **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of the page with the date the action occurred.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1007

**Title: Use of Instrument Records and Logbooks**

Revision history:

| Number | Date |
|---|---|
| 1.0 | 9/5/91 |
| 2 | 5/13/98 |
| 3 | 6/19/00 |
| 4 | 5/26/04 |
| 5 | 9/23/05 |
| 6 | 6/11/07 |

Revised by: Harry Romberg Date: 6/11/07
Harry Romberg, QA Officer

Approved by: Hugh Prentice Date: 6/11/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Introduction

1.1.1 The maintenance of instrument logbooks is essential to monitoring instrument performance and throughput and in tracking analyses. It is also important to confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. Several systems are in place at Laucks, the differences being primarily dependent on the specific instrument and analysis types. This SOP will discuss what is expected in each.

1.2 Scope

1.2.1 This SOP primarily addresses instrument run log maintenance, maintenance manuals and other logs not addressed in other SOPs. Standards logs, for instance, are discussed in the standards SOP, LTL-1013. Analytical balance logs are discussed in that SOP, LTL-1005. Cold storage logs are discussed in LTL-1006. Control and monitoring of logbooks and general items pertinent to all logbooks are discussed in Laucks SOP LTL-1019.

1.3 Definition of Terms

1.3.1 Logbook - Any bound, unbound or electronic document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs, standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs, or any other regularly maintained record of activity.

## 2. Equipment List and Standards

2.1 Equipment

2.1.1 maintenance logbook, analytical run logbook (where appropriate) or other applicable logbook

2.1.2 pen (pencil is NOT allowed)

## 3. Operation procedures

3.1 All Logbooks

3.1.1 All logbooks should be numbered and controlled according to procedures outlined in Laucks SOP LTL-1019. It is the analyst's responsibility before initiating any new

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**hardcopy** logbook to ensure that the logbook has been identified and given a logbook number by QA. See LTL-1019 for further detail.

3.1.2 **NOTE:** All errors in all **hardcopy** logbooks must be altered by a single-line cross out which must also be initialed and dated. No erasures, overwriting, white-out or multiple-line cross outs (blacking out) are acceptable.

3.1.3 **NOTE:** Empty space in **hardcopy** logbooks must be lined out (preferably with a Z for large blocks of empty space). This mark, as with error correction, should be initialed and dated.

3.2 Maintenance Manuals

3.2.1 All instruments at Laucks from GC or GC/MS systems to ICP or ICP/MS systems, spectrophotometers, ion chromatographs, etc. have instrument maintenance manuals associated with the specific instrument. For a number of reasons, it is extremely important that these logbooks be meticulously maintained.

- Problems and repeat problems and their resolution can be identified.
- The manual can be a source of ideas for troubleshooting instrument problems.
- The manual can be used to track the life of various parts.
- Notations in the manual should be used to document when instrument problems or failures have been resolved and that adequate routine instrument maintenance is occurring.

3.2.2 Maintenance manuals are bound notebooks with the specific instrument and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which may be the case for instruments which have been in service for a long time, especially if they have required extensive, ongoing maintenance, the notebooks should be clearly numbered on the cover as #1, #2, etc.

3.2.3 As a general rule, loose leaf or 3-ring bound notebooks are not acceptable. The exception to this rule is for maintaining copies of professional service call paperwork or if specific forms have been created for monitoring maintenance activities. Such paperwork must be dated in order to have a chronological record of maintenance activity that can be related to both the instrument history and sample analyses that occurred on an instrument both before and after any such activity. Note of the service should still be made in the bound notebook associated with that instrument and the identifying number on the service log noted in the maintenance manual.

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3.2.4 With a few basic rules, these maintenance manuals are free-form with no specific format but MUST include any and all maintenance associated with the particular instrument.

3.2.4.1 Each entry should be INITIALED by the person making the entry.

3.2.4.2 The maintenance manual must contain the DATE any service or maintenance was performed on the instrument and exactly WHAT that operation was. This includes everything from changing a part to cleaning an instrument orifice or changing a chromatographic column or instrument tubing. It should include everything from the simplest maintenance to the most complex, including any professional service calls.

3.2.4.3 Minimal space, usually not more than one line, should separate individual daily entries.

3.2.4.4 Where maintenance is routine, some books use codes for the most common service operations. These codes must be clearly defined either on the front, inside cover of the maintenance manual or on the first page. If there are multiple books, these codes must be so defined in EACH book.

3.2.5 If the maintenance was performed because of a specific problem (not just routine, ongoing maintenance) the problem should be described in at least one entry in the maintenance book as well as the work performed at any one time, and the outcome of that maintenance, that is whether or not it was successful or what occurred when the work was performed. Resolution of a malfunction should include not only that the service was successful but how it was demonstrated as having been so (i.e. the torch now lights, the oven came up to temperature, the baseline is now stable, etc.)

3.2.6 In order to aid in monitoring instrument performance changes, service or equipment changes may also be noted in instrument run logs. However, this information is supplementary. ALL maintenance must be recorded in the maintenance manual.

3.3 Instrument Run-Logs

3.3.1 Instrument run-logs come in two essentially different forms, with variations depending upon the specific instrument. In any form, a copy of the daily run log must accompany the data for any samples associated with that sequence.

3.3.2 Some run-logs are in bound, pre-printed, sequentially page-numbered books. They are identified by the specific instrument type and, if appropriate where multiple similar instruments are involved, instrument names or numbers printed on the outside cover. If there are multiple books for an instrument, which will be the case for instruments which have been in service for very long, the notebooks should be clearly numbered on the cover as #1, #2, etc.

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3.3.2.1 Run logs must identify the method being run either at the top of the page, or if more than one method is being used for any sequence, clearly marked by the sample entry. It is recognized that it is in some cases possible to use different methods, which may only be different in the way a calibration is interpreted or validated. It may even be that two methods are essentially identical. However, in these instances, the logsheet should clearly indicate for which method a particular sample is being analyzed.

3.3.2.2 Instrument run-logs should include places to record all relevant sample and data file IDs, performance criteria, sample type and size, additional comments pertinent to the specific analyses, and analyst initials. All appropriate information must be filled out and the page dated. Examples of a few current logbook forms (at the time of this writing) are located in Appendices I-IV. These forms should be considered examples and not as the only forms used by Laucks for this purpose. These forms may change with approval of the department manager and QA. Although this SOP will not then be considered invalid, new example forms should be incorporated into the next revision.

3.3.3 In addition to the appropriate header information for each analytical run, all of the pertinent information should be filled out for each injection.

3.3.4 The standards, samples, calibration checks, reference materials, QC samples, etc. should be listed in the order that they were analyzed.

3.3.5 Logbook information should be either completely filled out, or a logbook designed to incorporate all of the pertinent elements for that analysis so that all fields are filled in. Logbooks should contain all of the necessary information to track what analyses occurred, the processing order, and critical run parameters (such as what GC column was in use).

3.3.6 No empty space should be left between daily logbook entries. The end of the analytical sequence should be clearly marked and empty space on the page crossed out, the accepted practice being with a "Z" which covers the entire space being crossed out. This "Z" should be initialed and dated by the analyst making it.

3.3.7 The other type of run-log typically in use is the individual, loose-leaf instrument run-log printout. Where the instruments themselves don't produce such printouts, handwritten run-logs are produced by the analyst. These are the log types are most often in use in the Inorganics area of the laboratory but are disappearing as the flexibility of the LIMS allows better us of electronic forms.

3.3.8 A copy of the run-log is included with each data packet associated with that run.

3.3.9 As with the bound book format, the samples, standards, calibration checks, reference materials, etc. should be identified and listed IN ORDER.

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3.3.10 Information critical to identifying the analytical run (date, analyst, instrument, analysis type) must be included in the header information. If multiple analytical runs were made in one day, they must be identified as different runs by some clear identifying mechanism to indicate which run was which. This is inherent to the LIMS which makes it the best mechanism for maintaining the individual identity of the run. If the instrument is capable of time-stamping run data, this option should be utilized, although it need not be included in the run-log itself.

3.3.11 Where possible laboratory practice is to maintain ongoing run-logs for inorganic instrumentation. The daily run-logs are included with all data. Records which do not lend themselves to being kept in a pre-printed bound logbook may be collected in a 3-ring binder in an organized format but not unbound or loose-leaf. After sufficient logs have been collected, they should be bound with the laboratory comb binder. These logs should be given QA logbook IDs as described in Laucks SOP LTL-1019.

3.3.12 If an instrument produces a large quantity of data in a relatively short time, it may be easier to accumulate in date/time order in a banker's box which will be numbered and filed as an individual run log. Such data must be clearly dated and time-stamped as it is even more critical to maintaining correct order.

3.4 Other Logbooks

3.4.1 The same general principals used for the above logbooks apply to any other logbook, unless otherwise defined in a specific SOP.

3.4.1.1 Entries should be initialed and dated.

3.4.1.2 Empty space between entries should be minimized

3.4.1.3 Errors and empty spaces should be properly crossed out, initialed and dated.

3.4.1.4 Pages are preferably sequentially numbered but if this is not practical, at least dated and/or time stamped.

3.4.1.5 The logbook should identify the operation being monitored.

3.4.1.6 The pages in the logbook should contain all appropriate information needed to identify the activity and all applicable spaces should be completely filled out.

3.4.1.7 The logbook should be given a QA ID number as described in LTL-1019.

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## 4. References

*4.1.1* *National Environmental Laboratory Accreditation Conference, Ch. 5 Quality Systems, 6/5/2003, Section 5.4.12*

*4.1.2* *Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3,March 2005, Section 4.12*

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# Appendix I

## GC/MS Run Logs

Controlled Document

Book: 20 Assigned to: Tetra Tech

# Injection Log Summary Report

Method : U:\ZOOEY\QUANT\Z8270M.M (RTE Integrator)
Title : 8270 SW846 BNA Calibration 5970Z
Start (Tune) File ID : U:\ZOOEY\052004\Z0520001.D
Injection Date : 20 May 2004 Log Time Period (hrs) : ALL
Injection Time : 08:42 Total files within period : 15
Sample Directory : U:\ZOOEY\052004\

Injection Log Summary Table

| File ID | Multiplier I | S | T | Sample Name Misc Info | Date | Time |
|---|---|---|---|---|---|---|
| Z0520002 | 1.00 | 1.00 | 1.00 | SSTD010 5NG/UL ABN STD 5UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 09:53 |
| Z0520003 | 1.00 | 1.00 | 1.00 | SSTD020 10NG/UL ABN STD 10UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 10:26 |
| Z0520004 | 1.00 | 1.00 | 1.00 | SSTD050 25NG/UL ABN STD 25UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 10:58 |
| Z0520005 | 1.00 | 1.00 | 1.00 | SSTD080 40NG/UL ABN STD 40UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 11:31 |
| Z0520006 | 1.00 | 1.00 | 1.00 | SSTD120 60NG/UL ABN STD 60UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 12:04 |
| Z0520007 | 1.00 | 1.00 | 1.00 | SSTD160 80NG/UL ABN STD 80UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 12:36 |
| Z0520008 | 1.00 | 1.00 | 1.00 | ICV052004-1 1:5 MS9-16-15 + IS 5970Z | 20 May 2004 | 13:09 |
| Z0520009 | 1.00 | 1.00 | 1.00 | ICV052004-2 1:10 EX10-38-3 + IS 5970Z | 20 May 2004 | 13:42 |
| Z0520010 | 1.00 | 1.00 | 1.00 | CCV052004-1 25UL MS9-26-9/MS9-26-11 + | 20 May 2004 | 14:14 |
| Z0520011 | 1.00 | 1.00 | 1.00 | 0404492-06 CK SAMPLE 1:2 15G->0.5ML | 20 May 2004 | 14:47 |
| Z0520012 | 1.00 | 1.00 | 1.00 | B051904MSVWLS ARC29 1000ML->1.0ML + IS | 20 May 2004 | 15:19 |
| Z0520013 | 1.00 | 1.00 | 1.00 | S051904MSVWLS ARC29 1000ML->1.0ML + IS | 20 May 2004 | 15:52 |
| Z0520014 | 1.00 | 1.00 | 1.00 | 0405255-04 DMO-5-MWB ARC29 1050ML->1.0ML + IS | 20 May 2004 | 16:25 |
| Z0520015 | 1.00 | 1.00 | 1.00 | 0405255-05 DMO-5-MWA ARC29 1060ML->1.0ML + IS | 20 May 2004 | 16:58 |
| Z0520016 | 1.00 | 1.00 | 1.00 | RINSE | 20 May 2004 | 17:30 |

DFTTP : MS9-23-9 IS : MS9-26-12 RW 5/25/04

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Book: 20 Assigned to: Tetra Tech

# Injection Log Summary Report

Method : Q:\MSDCHEM\1\QUANT\8260B.M (RTE Integrator)
Title : VOA Standards for 5 point calibration 8260- 5ML
Start (Tune) File ID : K:\HPCHEM\1\DATA\052404\F0524001.D
Injection Date : 24 May 2004 Log Time Period (hrs) : ALL
Injection Time : 08:51 Total files within period : 25
Sample Directory : K:\HPCHEM\1\DATA\052404\

Injection Log Summary Table

| File ID | Multiplier I | S | T | Sample Name / Misc Info | Date | Time |
|---|---|---|---|---|---|---|
| F0524002 | 1.00 | 1.00 | 1.00 | I052404MWOF1<br>5G/5ML+IS/SS(MV8-16-14) | 24 May 2004 | 09:33 |
| F0524003 | 1.00 | 1.00 | 1.00 | BFB50NG<br>2UL(MV8-11-3) DIRECT INJ | 24 May 2004 | 10:11 |
| F0524004 | 1.00 | 1.00 | 1.00 | BFB50NG<br>2UL(MV8-11-3) DIRECT INJ | 24 May 2004 | 10:31 |
| F0524005 | 1.00 | 1.00 | 1.00 | BFB50NG<br>2UL(MV8-11-3) DIRECT INJ | 24 May 2004 | 10:50 |
| F0524006 | 1.00 | 1.00 | 1.00 | VSTD010<br>25ML+std(MV8-17-11)+IS/SS | 24 May 2004 | 11:21 |
| F0524007 | 1.00 | 1.00 | 1.00 | S052404MW0F1<br>25ML+IS/SS+MS(MV8-15-20) | 24 May 2004 | 12:00 |
| F0524008 | 1.00 | 1.00 | 1.00 | B052404MW0F1<br>25MLpfw+IS/SS(MV8-16-14) | 24 May 2004 | 12:34 |
| F0524009 | 1.00 | 1.00 | 1.00 | B052404MW0F2<br>25MLpfw+IS/SS(MV8-16-14) | 24 May 2004 | 13:08 |
| F0524010 | 1.00 | 1.00 | 1.00 | S052404MW0F2<br>25ML+IS/SS+MS(MV8-15-20) | 24 May 2004 | 13:42 |
| F0524011 | 1.00 | 1.00 | 1.00 | 0405247-01 KCSW<br>25ML+IS/SS | 24 May 2004 | 14:41 |
| F0524012 | 1.00 | 1.00 | 1.00 | 0405247-02 KCSW<br>25ML+IS/SS | 24 May 2004 | 15:15 |
| F0524013 | 1.00 | 1.00 | 1.00 | 0405247-03 KCSW<br>25ML+IS/SS | 24 May 2004 | 15:49 |
| F0524014 | 1.00 | 1.00 | 1.00 | 0405246-01 KCSW<br>25ML+IS/SS | 24 May 2004 | 16:23 |
| F0524015 | 1.00 | 1.00 | 1.00 | 0405246-02 KCSW<br>25ML+IS/SS | 24 May 2004 | 16:57 |
| F0524016 | 1.00 | 1.00 | 1.00 | 0405246-03 KCSW<br>25ML+IS/SS | 24 May 2004 | 17:31 |
| F0524017 | 1.00 | 1.00 | 1.00 | 0405268-01DL KCSW<br>1:100 25ML+IS/SS | 24 May 2004 | 18:06 |
| F0524018 | 1.00 | 1.00 | 1.00 | 0405268-02 KCSW<br>25ML+IS/SS | 24 May 2004 | 18:40 |
| F0524019 | 1.00 | 1.00 | 1.00 | 0405271-01 KCSW<br>25ML+IS/SS | 24 May 2004 | 19:14 |
| F0524020 | 1.00 | 1.00 | 1.00 | 0405271-02 KCSW<br>25ML+IS/SS | 24 May 2004 | 19:48 |
| F0524021 | 1.00 | 1.00 | 1.00 | 0405289-01 KCSW<br>25ML+IS/SS | 24 May 2004 | 20:22 |
| F0524022 | 1.00 | 1.00 | 1.00 | 0405289-02 KCSW<br>25ML+IS/SS | 24 May 2004 | 20:57 |
| F0524023 | 1.00 | 1.00 | 1.00 | 0405290-01 KCSW<br>25ML+IS/SS | 24 May 2004 | 21:31 |

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Book: 20 Assigned to: Tetra Tech

# Injection Log Summary Report

Method : Q:\MSDCHEM\1\QUANT\8260B.M (RTE Integrator)
Title : VOA Standards for 5 point calibration 8260- 5ML
Start (Tune) File ID : K:\HPCHEM\1\DATA\052404\F0524001.D
Injection Date : 24 May 2004 Log Time Period (hrs) : ALL
Injection Time : 08:51 Total files within period : 25
Sample Directory : K:\HPCHEM\1\DATA\052404\

Injection Log Summary Table

| File ID | Multiplier I | S | T | Sample Name Misc Info | Date | Time |
|---|---|---|---|---|---|---|
| F0524024 | 1.00 | 1.00 | 1.00 | 0405290-02 KCSW 25ML+IS/SS | 24 May 2004 | 22:05 |
| F0524025 | 1.00 | 1.00 | 1.00 | 0405291-01 KCSW 25ML+IS/SS | 24 May 2004 | 22:39 |
| F0524026 | 1.00 | 1.00 | 1.00 | 0405291-02 KCSW 25ML+IS/SS | 24 May 2004 | 23:13 |

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# Appendix II

## GC/VOA & HPLC Run Logs

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Book: 20 Assigned to: Tetra Tech

Laucks Testing Labs

# GC VOA Instrument Log

Analyst : _____ Date : _____

Calibration Reference : _____ Standard ID : _____
_____ Spike ID : _____
_____ Surrogate ID : _____

| File # | Sample ID | pH | Sample Vol / Wt | Std. / Spike / Surr Volume (ul) | | | Comments | Bottle # |
|---|---|---|---|---|---|---|---|---|
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Laucks Testing Laboratories, Inc.

BATCH SUMMARY ( \\TPER\Chek4\Pec3x.i\F51104syn.b )

| | Filename | Lab Sample ID | Client Sample ID | SDG | I.V. | D.F. | Inj. Date & Time |
|---|---|---|---|---|---|---|---|
| 1 | F5110402.D | STD01 50PPB | HPLC1620/1611 400X | | 0 | 1 | 11-MAY-2004 14:20 |
| 2 | F5110403.D | STD01 50PPB | HPLC1620/1611 400X | | 0 | 1 | 11-MAY-2004 14:57 |
| 3 | F5110404.D | STD02 100PPB | HPLC1620/1611 200X | | 0 | 1 | 11-MAY-2004 15:34 |
| 4 | F5110405.D | STD03 500PPB | HPLC1620/1611 40X | | 0 | 1 | 11-MAY-2004 16:11 |
| 5 | F5110406.D | STD04 1000PPB | HPLC1620/1611 20X | | 0 | 1 | 11-MAY-2004 16:48 |
| 6 | F5110407.D | STD05 5000PPB | HPLC1620/1611 4X | | 0 | 1 | 11-MAY-2004 17:25 |
| 7 | F5110408.D | IBLK 2000PPB | HPLC1-7-9 10X | | 0 | 1 | 11-MAY-2004 18:02 |
| 8 | F5110409.D | ICV 2000PPB | HPLC1-7-1 10X | | 0 | 1 | 11-MAY-2004 18:39 |
| 9 | F5110410.D | STD04 1000PPB | HPLC1-6-20 20X | | 0 | 1 | 11-MAY-2004 19:17 |
| 10 | F5110411.D | B042804HORSMS | B042804HORSMS | 31108 | 0 | 1 | 11-MAY-2004 19:54 |
| 11 | F5110412.D | 0404313-01 | 03SS1140002 | 31108 | 0 | 1 | 11-MAY-2004 20:31 |
| 12 | F5110413.D | 0404313-03 | 03SS0880002 | 31108 | 0 | 1 | 11-MAY-2004 21:08 |
| 13 | F5110414.D | 0404313-03DL | 03SS0880002DL | 31108 | 0 | 20 | 11-MAY-2004 21:45 |
| 14 | F5110415.D | 0404313-04 | 03SB0880206 | 31108 | 0 | 1 | 11-MAY-2004 22:22 |
| 15 | F5110416.D | 0404313-05 | 03SS0890002 | 31108 | 0 | 1 | 11-MAY-2004 22:59 |
| 16 | F5110417.D | 0404313-06 | 03SB0890206 | 31108 | 0 | 1 | 11-MAY-2004 23:36 |
| 17 | F5110418.D | 0404313-07 | 03SB0950206 | 31108 | 0 | 1 | 12-MAY-2004 00:13 |
| 18 | F5110419.D | 0404313-10 | 03SS0980002 | 31108 | 0 | 1 | 12-MAY-2004 00:50 |
| 19 | F5110420.D | 0404313-11 | 03SB0980206 | 31108 | 0 | 1 | 12-MAY-2004 01:27 |
| 20 | F5110421.D | STD04 1000PPB | HPLC1-6-20 20X | | 0 | 1 | 12-MAY-2004 02:04 |
| 21 | F5110422.D | B043004HORSMO | B043004HORSMO | 31112 | 0 | 1 | 12-MAY-2004 02:41 |
| 22 | F5110423.D | 0404383-01 | 03SS1150002 | 31112 | 0 | 1 | 12-MAY-2004 03:18 |
| 23 | F5110424.D | 0404383-03 | 03SS1170002 | 31112 | 0 | 1 | 12-MAY-2004 03:55 |
| 24 | F5110425.D | 0404383-07 | 03SS1190002 | 31112 | 0 | 1 | 12-MAY-2004 04:32 |
| 25 | F5110426.D | 0404383-07DL | 03SS1190002DL | 31112 | 0 | 10 | 12-MAY-2004 05:09 |
| 26 | F5110427.D | 0404383-08 | 03SB1190206 | 31112 | 0 | 1 | 12-MAY-2004 05:46 |
| 27 | F5110428.D | 0404383-09 | 03SS1210002 | 31112 | 0 | 1 | 12-MAY-2004 06:23 |
| 28 | F5110429.D | 0404383-09DL | 03SS1210002DL | 31112 | 0 | 5 | 12-MAY-2004 07:00 |

I.V. = Inj.Vol. per Column  Batch Summary  Ver 2.0 9-5-2002

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Book: 20 Assigned to: Tetra Tech



# Appendix III

## GPC Run Log

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Book: 20 Assigned to: Tetra Tech

# Laucks Testing Labs, Inc.
## GPC Bench Sheet



| | |
|---|---|
| Date | 5-5-04 |
| Cal. Ref | 050304GPC2 |
| Analyst | AGH |
| Case # | |
| SDG # | ATP01 |
| Matrix | Soil |
| Program | BAN (1) |
| Load Time | 0:20.0 |
| Dump Time | 26:44 |
| Collect Time | 26:26 |
| Wash Time | 16:38 |

Operating Conditions:

| | | |
|---|---|---|
| N2 Pressure | — | |
| tank, high | 150 | psi |
| tank, low | 39 | psi |
| line | 30 | psi |
| Rinse Press. | 12 | psi |
| Sys. Press. | 13 | psi |
| Flow Rate | 5.0 | ml/min |
| Temp | 70 | deg F |
| Chart Speed | 20.0 | cm/~~min~~ hr |
| Chart Full Scale | 0.01 | Volts |
| UV Detector | 0.5 | AUFS |
| Column ID | A2274-17 | |

| Port | LTL Number | Client ID | Intermed Vol (ml) | Aliquot Clean (ml) | Volume Collect (ml) | Final Vol (ml) |
|---|---|---|---|---|---|---|
| 1 | 0404524-B050404MSVSLY | | 10.0 | 5.0 | 130 | 0.5 |
| 2 | -S050404MSVSLY | | | | | |
| 3 | -02 | | | | | |
| 4 | -04 | | | | | |
| 5 | -05 | | | | | |
| 6 | -06 | | | | | |
| 7 | -13 | | | | | |
| 8 | -13MS | | | | | |
| 9 | -13MSD | | | | | |
| 10 | -15 | | | | | |
| 11 | | | | | | |
| 12 | | | | | | |
| 13 | | | | | | |
| 14 | | | | | | |
| 15 | | | | | | |
| 16 | | | | 5-5-04 | | |
| 17 | | | | | | |
| 18 | | | AGH | | | |
| 19 | | | | | | |
| 20 | | | | | | |
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| 22 | | | | | | |
| 23 | | | | | | |

Comments: $MeCl_2$ (B# 5 #C5842), Filter Lot # 5252

Controlled Document
Book: 20 Assigned to: Tetra Tech



# Appendix IV

## Ion Chromatography Run Logs

Controlled Document

DIONEX SCHEDULE - C:\DX\SCHEDULE\IC060507.SCH

Book: 20 Assigned to: Tetra Tech

| Inj# | Sample Name | Method | Data File | Vol. | Dil. | Int.Std. |
|---|---|---|---|---|---|---|
| 1 | RINSE | ..\IC060507 | ..\IC060501.D01 | 1 | 1 | 1 |
| 2 | ICV/SRM1(25uL IC-7- | ..\IC060507 | ..\IC060501.D02 | 1 | 1 | 1 |
| 3 | ICB/B060507IAIW01 | ..\IC060507 | ..\IC060501.D03 | 1 | 1 | 1 |
| 4 | SRM2 | ..\IC060507 | ..\IC060501.D04 | 1 | 1 | 1 |
| 5 | B060507IAIW02 | ..\IC060507 | ..\IC060501.D05 | 1 | 1 | 1 |
| 6 | COFO070601-001 | ..\IC060507 | ..\IC060501.D06 | 1 | 1 | 1 |
| 7 | COFO070601-001DL 10 | ..\IC060507 | ..\IC060501.D07 | 1 | 1 | 1 |
| 8 | COFO070601-002 | ..\IC060507 | ..\IC060501.D08 | 1 | 1 | 1 |
| 9 | COFO070601-002DL 10 | ..\IC060507 | ..\IC060501.D09 | 1 | 1 | 1 |
| 10 | COFO070601-003 | ..\IC060507 | ..\IC060501.D10 | 1 | 1 | 1 |
| 11 | COFO070601-003DL 10 | ..\IC060507 | ..\IC060501.D11 | 1 | 1 | 1 |
| 12 | COFO070601-004 | ..\IC060507 | ..\IC060501.D12 | 1 | 1 | 1 |
| 13 | NSCH070601-001 | ..\IC060507 | ..\IC060501.D13 | 1 | 1 | 1 |
| 14 | CCV1(250uL IC-7-24- | ..\IC060507 | ..\IC060501.D14 | 1 | 1 | 1 |
| 15 | CCB1 | ..\IC060507 | ..\IC060501.D15 | 1 | 1 | 1 |
| 16 | NSCH070601-001DL 10 | ..\IC060507 | ..\IC060501.D16 | 1 | 1 | 1 |
| 17 | NSCH070601-002 | ..\IC060507 | ..\IC060501.D17 | 1 | 1 | 1 |
| 18 | RINSE | ..\IC060507 | ..\IC060501.D18 | 1 | 1 | 1 |
| 19 | NSCH070601-002DL 25 | ..\IC060507 | ..\IC060501.D19 | 1 | 1 | 1 |
| 20 | BCH31-002DL 50X | ..\IC060507 | ..\IC060501.D20 | 1 | 1 | 1 |
| 21 | BCH31-004DL 25X | ..\IC060507 | ..\IC060501.D21 | 1 | 1 | 1 |
| 22 | BCH31-007DL 50X | ..\IC060507 | ..\IC060501.D22 | 1 | 1 | 1 |
| 23 | BCH31-007MS 50X | ..\IC060507 | ..\IC060501.D23 | 1 | 1 | 1 |
| 24 | BCH31-007MSD 50X | ..\IC060507 | ..\IC060501.D24 | 1 | 1 | 1 |
| 25 | BCH31-008DL 20X | ..\IC060507 | ..\IC060501.D25 | 1 | 1 | 1 |
| 26 | CCV2 | ..\IC060507 | ..\IC060501.D26 | 1 | 1 | 1 |
| 27 | CCB2 | ..\IC060507 | ..\IC060501.D27 | 1 | 1 | 1 |
| 28 | BCH31-009DL 20X | ..\IC060507 | ..\IC060501.D28 | 1 | 1 | 1 |
| 29 | BCH31-010DL 20X | ..\IC060507 | ..\IC060501.D29 | 1 | 1 | 1 |
| 30 | BCH31-011DL 20X | ..\IC060507 | ..\IC060501.D30 | 1 | 1 | 1 |
| 31 | KSTS01-001DL 10X | ..\IC060507 | ..\IC060501.D31 | 1 | 1 | 1 |
| 32 | KSTS01-002DL 10X | ..\IC060507 | ..\IC060501.D32 | 1 | 1 | 1 |
| 33 | KSTS01-003DL 10X | ..\IC060507 | ..\IC060501.D33 | 1 | 1 | 1 |
| 34 | KSTS01-004DL 10X | ..\IC060507 | ..\IC060501.D34 | 1 | 1 | 1 |
| 35 | KSTS01-005DL 10X | ..\IC060507 | ..\IC060501.D35 | 1 | 1 | 1 |
| 36 | KSTS01-006DL 10X | ..\IC060507 | ..\IC060501.D36 | 1 | 1 | 1 |
| 37 | KSTS01-007DL 10X | ..\IC060507 | ..\IC060501.D37 | 1 | 1 | 1 |
| 38 | CCV3 | ..\IC060507 | ..\IC060501.D38 | 1 | 1 | 1 |
| 39 | CCB3 | ..\IC060507 | ..\IC060501.D39 | 1 | 1 | 1 |
| 40 | KSTS01-007MS 20X | ..\IC060507 | ..\IC060501.D40 | 1 | 1 | 1 |
| 41 | KSTS01-007MSD 20X | ..\IC060507 | ..\IC060501.D41 | 1 | 1 | 1 |
| 42 | BS | ..\IC060507 | ..\IC060501.D42 | 1 | 1 | 1 |
| 43 | BSD | ..\IC060507 | ..\IC060501.D43 | 1 | 1 | 1 |
| 44 | CCV4 | ..\IC060507 | ..\IC060501.D44 | 1 | 1 | 1 |
| 45 | CCB4 | ..\IC060507 | ..\IC060501.D45 | 1 | 1 | 1 |
| 46 | | ..\SHUTDOWN | | 1 | 1 | 1 |

Comment:

Controlled Document
Book: 20 Assigned to: Tetra Tech

R018378



Sequence: 060507P
Operator: colleenc
Printed: 6/6/2007 10:48:54 AM

Title:
Datasource: D33TPG41_local
Location:
Timebase: PERC
#Samples: 29

Created: 6/5/2007 1:25:10 PM by colleenc
Last Update: 6/5/2007 1:26:55 PM by colleenc

| No. | Name | Comment |
|---|---|---|
| 1 | R | ICS-2500 DIONEX B |
| 2 | R | ICS-2500 DIONEX B |
| 3 | std1 1ppb | ICS-2500 DIONEX B (200 uL IC-7-24-10) |
| 4 | std2 2ppb | ICS-2500 DIONEX B (400 uL IC-7-24-10) |
| 5 | std3 5ppb | ICS-2500 DIONEX B (1000 uL IC-7-24-10) |
| 6 | std4 10ppb | ICS-2500 DIONEX B (2000 uL IC-7-24-10) |
| 7 | std5 25ppb | ICS-2500 DIONEX B (5 mL IC-7-24-10) |
| 8 | std6 80ppb | ICS-2500 DIONEX B (1000 uL IC-7-24-9) |
| 9 | RINSE | ICS-2500 DIONEX B |
| 10 | MCT/IPC | ICS-2500 DIONEX B (5 mL IC-7-24-7) |
| 11 | ICV | ICS-2500 DIONEX B (2500 uL IC-7-24-8) |
| 12 | ICB | ICS-2500 DIONEX B |
| 13 | BLK SPK - SAND | ICS-2500 DIONEX B (250 uL IC-7-24-9) |
| 14 | BLK SPK DUP - SAND | ICS-2500 DIONEX B (250 uL IC-7-24-9) |
| 15 | B060507PERS01 | ICS-2500 DIONEX B |
| 16 | 1 ppb CHECK STD | ICS-2500 DIONEX B (200 uL IC-7-24-10) |
| 17 | PCGW070501-016 | ICS-2500 DIONEX B |
| 18 | PCGW070501-017 | ICS-2500 DIONEX B |
| 19 | PCGW070501-018 | ICS-2500 DIONEX B |
| 20 | PCGW070501-019 | ICS-2500 DIONEX B |
| 21 | PCGW070501-020 | ICS-2500 DIONEX B |
| 22 | PCGW070501-021 | ICS-2500 DIONEX B |
| 23 | CCV1 | ICS-2500 DIONEX B (2000 uL IC-7-24-10) |
| 24 | CCB1 | ICS-2500 DIONEX B |
| 25 | PCGW070501-022 | ICS-2500 DIONEX B |
| 26 | PCGW070501-023 | ICS-2500 DIONEX B |
| 27 | CCV2 | ICS-2500 DIONEX B (2000 uL IC-7-24-10) |
| 28 | CCB2 | ICS-2500 DIONEX B |
| 29 | SHUTDOWN | ICS-2500 DIONEX B |

Controlled Document
Book: 20 Assigned to: Tetra Tech



Sequence: 060507P
Operator: colleenc
Printed: 6/6/2007 10:48:54 AM

Title:
Datasource: D33TPG41_local
Location:
Timebase: PERC
#Samples: 29

Created: 6/5/2007 1:25:10 PM by colleenc
Last Update: 6/5/2007 1:26:55 PM by colleenc

| No. | Name | Type | Pos. | Inj. Vol. | Program | Method | Status | Inj. Date/Time |
|---|---|---|---|---|---|---|---|---|
| 1 | R | Unknown | 1 | 5.0 | perchlorate | perc | Finished | 6/5/2007 4:42:47 PM |
| 2 | R | Unknown | 2 | 5.0 | perchlorate | perc | Finished | 6/5/2007 5:14:12 PM |
| 3 | std1 1ppb | Standard | 3 | 5.0 | perchlorate | perc | Finished | 6/5/2007 5:45:37 PM |
| 4 | std2 2ppb | Standard | 4 | 5.0 | perchlorate | perc | Finished | 6/5/2007 6:17:02 PM |
| 5 | std3 5ppb | Standard | 5 | 5.0 | perchlorate | perc | Finished | 6/5/2007 6:48:27 PM |
| 6 | std4 10ppb | Standard | 6 | 5.0 | perchlorate | perc | Finished | 6/5/2007 7:19:53 PM |
| 7 | std5 25ppb | Standard | 7 | 5.0 | perchlorate | perc | Finished | 6/5/2007 7:51:18 PM |
| 8 | std6 80ppb | Standard | 8 | 5.0 | perchlorate | perc | Finished | 6/5/2007 8:22:43 PM |
| 9 | RINSE | Unknown | 9 | 5.0 | perchlorate | perc | Finished | 6/5/2007 8:54:08 PM |
| 10 | MCT/IPC | Unknown | 10 | 5.0 | perchlorate | perc | Finished | 6/5/2007 9:25:34 PM |
| 11 | ICV | Unknown | 11 | 5.0 | perchlorate | perc | Finished | 6/5/2007 9:56:59 PM |
| 12 | ICB | Unknown | 12 | 5.0 | perchlorate | perc | Finished | 6/5/2007 10:28:24 PM |
| 13 | BLK SPK - SAND | Unknown | 13 | 5.0 | perchlorate | perc | Finished | 6/5/2007 10:59:49 PM |
| 14 | BLK SPK DUP - SAND | Unknown | 14 | 5.0 | perchlorate | perc | Finished | 6/5/2007 11:31:14 PM |
| 15 | B060507PERS01 | Unknown | 15 | 5.0 | perchlorate | perc | Finished | 6/6/2007 12:02:39 AM |
| 16 | 1 ppb CHECK STD | Unknown | 16 | 5.0 | perchlorate | perc | Finished | 6/6/2007 12:34:04 AM |
| 17 | PCGW070501-016 | Unknown | 17 | 5.0 | perchlorate | perc | Finished | 6/6/2007 1:05:29 AM |
| 18 | PCGW070501-017 | Unknown | 18 | 5.0 | perchlorate | perc | Finished | 6/6/2007 1:36:54 AM |
| 19 | PCGW070501-018 | Unknown | 19 | 5.0 | perchlorate | perc | Finished | 6/6/2007 2:08:19 AM |
| 20 | PCGW070501-019 | Unknown | 20 | 5.0 | perchlorate | perc | Finished | 6/6/2007 2:39:44 AM |
| 21 | PCGW070501-020 | Unknown | 21 | 5.0 | perchlorate | perc | Finished | 6/6/2007 3:11:10 AM |
| 22 | PCGW070501-021 | Unknown | 22 | 5.0 | perchlorate | perc | Finished | 6/6/2007 3:42:35 AM |
| 23 | CCV1 | Unknown | 23 | 5.0 | perchlorate | perc | Finished | 6/6/2007 4:14:03 AM |
| 24 | CCB1 | Unknown | 24 | 5.0 | perchlorate | perc | Finished | 6/6/2007 4:45:29 AM |
| 25 | PCGW070501-022 | Unknown | 25 | 5.0 | perchlorate | perc | Finished | 6/6/2007 5:16:54 AM |
| 26 | PCGW070501-023 | Unknown | 26 | 5.0 | perchlorate | perc | Finished | 6/6/2007 5:48:19 AM |
| 27 | CCV2 | Unknown | 27 | 5.0 | perchlorate | perc | Finished | 6/6/2007 6:19:45 AM |
| 28 | CCB2 | Unknown | 28 | 5.0 | perchlorate | perc | Finished | 6/6/2007 6:51:10 AM |
| 29 | SHUTDOWN | Unknown | 29 | 5.0 | shutdown | perc | Finished | 6/6/2007 7:22:35 AM |

Controlled Document
Book: 20 Assigned to: Tetra Tech



Sequence: 060507P
Operator: colleen2
Printed: 6/6/2007 10:48:54 AM

Title:
Datasource: D33TPG41_local
Location:
Timebase: PERC
#Samples: 29

Created: 6/5/2007 1:25:10 PM by colleenc
Last Update: 6/5/2007 1:26:55 PM by colleenc

| No. | Name | Weight | Dil. Factor | ISTD Amount | Sample ID | Replicate ID |
|---|---|---|---|---|---|---|
| 1 | R | 1.0000 | 1.0000 | 1.0000 | | |
| 2 | R | 1.0000 | 1.0000 | 1.0000 | | |
| 3 | std1 1ppb | 1.0000 | 1.0000 | 1.0000 | | |
| 4 | std2 2ppb | 1.0000 | 1.0000 | 1.0000 | | |
| 5 | std3 5ppb | 1.0000 | 1.0000 | 1.0000 | | |
| 6 | std4 10ppb | 1.0000 | 1.0000 | 1.0000 | | |
| 7 | std5 25ppb | 1.0000 | 1.0000 | 1.0000 | | |
| 8 | std6 80ppb | 1.0000 | 1.0000 | 1.0000 | | |
| 9 | RINSE | 1.0000 | 1.0000 | 1.0000 | | |
| 10 | MCT/IPC | 1.0000 | 1.0000 | 1.0000 | | |
| 11 | ICV | 1.0000 | 1.0000 | 1.0000 | | |
| 12 | ICB | 1.0000 | 1.0000 | 1.0000 | | |
| 13 | BLK SPK - SAND | 1.0000 | 1.0000 | 1.0000 | | |
| 14 | BLK SPK DUP - SAND | 1.0000 | 1.0000 | 1.0000 | | |
| 15 | B060507PERS01 | 1.0000 | 1.0000 | 1.0000 | | |
| 16 | 1 ppb CHECK STD | 1.0000 | 1.0000 | 1.0000 | | |
| 17 | PCGW070501-016 | 1.0000 | 1.0000 | 1.0000 | | |
| 18 | PCGW070501-017 | 1.0000 | 1.0000 | 1.0000 | | |
| 19 | PCGW070501-018 | 1.0000 | 1.0000 | 1.0000 | | |
| 20 | PCGW070501-019 | 1.0000 | 1.0000 | 1.0000 | | |
| 21 | PCGW070501-020 | 1.0000 | 1.0000 | 1.0000 | | |
| 22 | PCGW070501-021 | 1.0000 | 1.0000 | 1.0000 | | |
| 23 | CCV1 | 1.0000 | 1.0000 | 1.0000 | | |
| 24 | CCB1 | 1.0000 | 1.0000 | 1.0000 | | |
| 25 | PCGW070501-022 | 1.0000 | 1.0000 | 1.0000 | | |
| 26 | PCGW070501-023 | 1.0000 | 1.0000 | 1.0000 | | |
| 27 | CCV2 | 1.0000 | 1.0000 | 1.0000 | | |
| 28 | CCB2 | 1.0000 | 1.0000 | 1.0000 | | |
| 29 | SHUTDOWN | 1.0000 | 1.0000 | 1.0000 | | |

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# Appendix IV

## Astoria 2 Run Log

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Run Name: 060607nnw01
Configuration: NO3/NO2
Run date: 6/6/2007

Laucks Testing Labs, Inc
Astoria 2 Autoanalyzer
Rachel Frank

SRM = 5 mL AP-37-11
NO3 Spike = AP-39-14
EN3 = 100 uL NO3 Spike
EN2 = 2 mL AP-39-15
Int Std = AP-41-12(CCV = 2 mL)

| | Identifier | Comment | Time | NO3/NO2 Raw Ht | NO3/NO2 mg/L |
|---|---|---|---|---|---|
| 1 | SYNC | | 8:14:33 AM | 0.2821 | 2.4296 |
| 2 | W | | 8:14:58 AM | 0.0030 | 0.0040 |
| 3 | w | | 8:16:14 AM | 0.0022 | -0.0049 |
| 4 | C1 | | 8:17:28 AM | 0.0023 | -0.0054 |
| 5 | C2 | 100 ul Int Std | 8:18:43 AM | 0.0084 | 0.0451 |
| 6 | C3 | 200 ul Int Std | 8:19:58 AM | 0.0149 | 0.1000 |
| 7 | C4 | 1 mL Int Std | 8:21:13 AM | 0.0629 | 0.5158 |
| 8 | C5 | 2 mL Int Std | 8:22:28 AM | 0.1184 | 0.9961 |
| 9 | C6 | 5 mL Int Std | 8:23:43 AM | 0.2914 | 2.4985 |
| | | | | | |
| | Curve #: | | | | 1 |
| | Curve Type: | | | | Linear |
| | Correlation: | | | | 0.99997 |
| | Intercept: | | | | -0.00045926 |
| | Linear coef: | | | | 0.11504 |
| | | | | | |
| 10 | W | | 8:24:58 AM | 0.0046 | 0.0040 |
| 11 | w | | 8:26:13 AM | 0.0040 | -0.0014 |
| 12 | ICV/SRM1 | | 8:27:29 AM | 0.1610 | 1.3637 |
| 13 | ICB | | 8:28:43 AM | 0.0046 | 0.0036 |
| 14 | MDL | | 8:29:58 AM | 0.0072 | 0.0262 |
| 15 | EN3 | | 8:31:13 AM | 0.1184 | 0.9928 |
| 16 | EN2 | | 8:32:28 AM | 0.1188 | 0.9967 |
| 17 | B060607NNW01 | | 8:33:43 AM | 0.0052 | 0.0089 |
| 18 | KC180702-001 | | 8:34:58 AM | 0.0111 | 0.0601 |
| ~~19~~ | ~~KC440701-001~~ | | ~~8:36:13 AM~~ | ~~0.3099~~ | ~~2.6573~~ |
| 20 | KC440701-003 | | 8:37:28 AM | 0.0214 | 0.1500 |
| 21 | KC440701-005 | | 8:38:43 AM | 0.0710 | 0.5811 |
| 22 | KC170703-001 | | 8:39:58 AM | 0.2173 | 1.8531 |
| 23 | KC020737-001 | | 8:41:13 AM | 0.0091 | 0.0428 |
| 24 | CCV1 | | 8:42:28 AM | 0.1219 | 1.0231 |
| 25 | CCB1 | | 8:43:43 AM | 0.0069 | 0.0237 |
| ~~26~~ | ~~KC020737-001MS~~ | | ~~8:44:58 AM~~ | ~~0.1159~~ | ~~0.9712~~ |
| 27 | KC020737-001MSD | | 8:46:13 AM | 0.1143 | 0.9572 |
| 28 | KC020737-002 | | 8:47:28 AM | 0.1291 | 1.0863 |
| 29 | KC020737-003 | | 8:48:43 AM | 0.0637 | 0.5177 |
| ~~30~~ | ~~KC020737-005~~ | | ~~8:49:58 AM~~ | ~~0.2303~~ | ~~1.9659~~ |

RL 6/6/07

RL 6/6/07

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Book: 20 Assigned to: Tetra Tech

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1008

Title: **QC Corrective Action**

Revision history:

| Number | Date |
|---|---|
| 1 (LTL-0008) | 12/12/86 |
| 2 (LTL-0008) | 5/19/87 |
| 3 (LTL-0008) | 6/29/89 |
| 4 | 3/3/96 |
| 5 | 6/22/96 |
| 6 | 6/16/00 |
| 7 | 3/16/05 |
| 8 | 7/13/07 |

Written by: Harry Romberg Date: 7/13/07
Harry Romberg, Quality Assurance Officer

Approved by: Hugh Prentice Date: 8/20/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to describe Laucks system of identifying, documenting and resolving out-of-control events and the actions taken to correct problems or issues that arise which may affect data quality or good laboratory practices.

1.2 Scope

1.2.1 The need for corrective action may be recognized by any member of Laucks staff. When issues arise that require corrective action, the analyst, supervisor, project manager, Lab Director and/or Quality Assurance work jointly, depending on the circumstances, to solve and correct the problem. Documentation is a key part of this process because it provides a way to track a problem and its resolution. This is useful in demonstrating that actions were taken, tracking to make sure the issues were resolved, and for future reference to help solve similar problems.

1.2.2 The need for corrective action, or at least the identification of non-conforming events, can arise from any aspect of laboratory operation. This may include the analytical process itself or any supporting activities such as sample receipt and storage, equipment function or monitoring as well as report and EDD production.

1.2.3 Depending on the nature of the problem, issues are documented using the Issues module [Help][Report a Problem] from the LIMS, on QC Outlier reports or in a few selected instances, on a logsheet with space specifically for such actions. Corrective action resulting from an audit is also dealt with using its own Audit Response form but at the present time, this action is elucidated in an SOP specific to that process.

1.2.4 Much of this documentation system, particularly as it relates to the LIMS, is relatively new at the time of this writing and changes may occur as needed to accommodate this process as it continues to develop.

## 2. Definition of Terms

2.1 This section defines terms and acronyms as they are used in this SOP.

2.1.1 **Corrective Action**: Action taken by an individual(s) to correct a problem as evidenced by either the failure of QC criteria or a more general problem which could affect performance of an analysis, the quality of service or other activity undertaken by the laboratory.

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2.1.2 **Out-of-control event**: Any occurrence or condition failing to meet Laucks QC criteria or has the potential to impact data quality.

2.1.3 **QA/QC**: Quality Assurance/Quality Control

2.1.4 **QAP**: Quality Assurance Plan – The laboratory document governing overall Quality Assurance (QA) policy and guidelines. This document discusses physical assets as well as administrative aspects of the laboratory's QA program.

2.1.5 **Reagent blank**: a measured volume of reagents used in a method upon which an analysis is performed in order to determine contamination or reagent contribution to the analytical results of the associated samples.

2.1.6 **RSD**: Relative Standard Deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values to one another.

2.1.7 **Method blank or Preparation Blank**: a reagent blank that undergoes a preparation (digestion, extraction, distillation, etc.) step prior to analysis.

2.1.8 **RPD**: Relative Percent Difference

2.1.9 **LCS**: Laboratory Control Sample

## 3. QC Outlier Procedure

3.1 Identifying a QC Outlier

3.1.1 The following is a list of examples of QC outliers. This is not a complete list of all possible outliers and many of those listed may be different for different methods. Specific criteria are given in analytical SOPs or in other QA documents. If there is doubt about whether a situation is out-of-control and must be responded to, consult with Quality Assurance.

3.1.1.1 GC/MS instrument tune criteria failing to meet criteria

3.1.1.2 Initial calibration linearity, depending upon the method used for calibration, correlation coefficient <0.995 (<0.990 for some fuels analyses) or percent RSD failing to meet method specifications.

3.1.1.3 Daily and continuing calibration verification or calibration blanks outside acceptable ranges as defined in their respective SOPs (either recovery or, in the case of chromatography, also the retention time).

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3.1.1.4 **NOTE:** If any of the above instances (3.1.1.1-3.1.1.3) occurs, analysis is stopped. No sample analysis can occur until the event is back in control. A QC Outlier Report form does not need to be filled out for these instances if identified at the analyst level and corrected before any sample data are generated. Since even sample data with non-compliant QC are sometimes used to support other results even if the non-compliant data itself is not reportable, once actual samples or sample-specific QC have been analyzed, the above (3.1.1.1 – 3.1.1.3) constitute out-of-control events and must be formally addressed using the appropriate forms and discussed in the case narrative of the affected SDG(s).

3.1.1.5 Matrix spike, surrogate spike or blank spike recoveries outside acceptable ranges.

3.1.1.6 Unacceptable RPD value for MS/MSD or duplicate samples.

3.1.1.7 Unacceptable values for LCS's and QC samples.

3.1.1.8 A method (reagent) blank containing interference or a target analyte at a concentration greater than or equal to ½ the method **reporting** limit.

**Note:** Results for samples which contain target analyte levels which are greater than 20 times the blank or which contain none of the offending analyte may be considered acceptable but still require a documented decision.

3.1.1.9 A sample received, prepared or analyzed past holding time. Even if the event is out of the laboratory's control, such as a sample received after the holding time had already expired, requires documentation, particularly if analyses are still conducted.

3.1.1.10 A sample depleted before all required analyses are completed.

3.1.1.11 An extract blown down to dryness, spilled or otherwise compromised.

3.1.1.12 Contaminated reagents and glassware.

3.1.1.13 Equipment malfunction or instrument failure, such as cold storage unit temperature outside acceptable ranges and the loss of data acquisition.

3.1.1.14 Record keeping omissions, errors, and deviations from the record keeping standard operating procedures are also out-of-control situations

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3.2 Responding to QC Outliers

3.2.1 When an out-of-control event is recognized, each individual involved with the analysis in question has an interactive role and responsibility, these are as follows:

3.2.2 Analyst:

3.2.2.1 Must be able to recognize QC failure and immediately take the proper action or, if unsure of the appropriate response, notify the supervisor and work with the supervisor and Quality Assurance to solve the problem.

3.2.2.2 Maintains QC control charts.

3.2.2.2.1 Protocols are being developed which will automate the production of control charts. However, it still must be the analyst who will take action for exceptions.

3.2.2.3 The analyst is also responsible for performing the following steps to correct the problem:

3.2.2.3.1 Examine all calculations for correctness

3.2.2.3.2 Examine bench sheets for correctness

3.2.2.3.3 Check instrumentation and operating conditions to preclude the possibility of malfunctions or operator error

3.2.2.3.4 Verify integrity of spiking solution, laboratory control sample, or calibration standard

3.2.2.3.5 Re-analyze the sample

3.2.2.3.6 Take other actions as noted in the specific analytical SOP.

3.2.2.3.7 If these steps do not yield acceptable results, consult the supervisor.

3.2.3 Supervisor:

3.2.3.1 Must review all analytical and QC data for reasonableness, accuracy and clerical errors; also responsible for QC control charts. Some of the above duties may be assigned to others, with supervisory oversight, if those others have been trained to observe the conditions which would initiate further investigation.

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3.2.3.2 In an out-of-control event, the supervisor works with the analyst and Quality Assurance to solve the problem and prevents the reporting of suspect data by stopping work on the analysis in question and insuring that analyses for all suspect results are repeated, if possible, after the source of the error is determined and remedied.

3.2.3.3 If corrective actions do not yield results that meet specifications, it may be determined that sufficient action has nevertheless been taken or that nothing can be done which will improve the data quality. The supervisor and QA will approve of such decisions and if it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client. In cases where the data are substantially impacted or entirely unusable, it may be necessary to contact the client (usually done by the Project Manager).

3.2.4 Quality Assurance:

3.2.4.1 The Quality Assurance Officer or designee will work with supervisory personnel and/or analysts to solve out-of-control situations which are not routinely corrected at the bench.

3.2.4.2 In the event that an out-of-control situation occurs that is unnoticed at the bench or supervisory level, such as performance failure on a blind QC sample, Quality Assurance will notify the supervisor, help identify and solve the problem where applicable, insuring the work is stopped on the analysis and no suspect data is reported.

3.2.4.3 Finally the Quality Assurance Officer or designee must review and approve all corrective action reports which cannot be resolved. If corrective actions do not yield results which meet specifications, it may be determined that sufficient action has nevertheless been taken or that nothing can be done which will improve the data quality. The supervisor and QA will approve of such decisions which are generally communicated to the client through the laboratory Project Manager.

3.2.4.4 If it is determined that the data quality could be impacted, the supervisor will ensure that appropriate comments are reported with the data to the client and QA will review said comments.

3.2.5 Project Manager:

3.2.5.1 The Project Manager is responsible for notifying the client of out-of-control events, such as missed holding times, raised reporting limits, matrix interferences, etc. which cannot be resolved without potential impact on either

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the data quality, the agreed upon or routinely reported results, or the timely and expected delivery date. It is not necessary to contact the client for events which are correctable and do not impact the final data quality, holding times or turn-around unless specifically requested by the client. These events may still be commented on in the report narrative, however.

3.3 Corrective Actions

3.3.1 Appropriate corrective action depends on the nature of the problem. The type of analysis, the extent of the discrepancy, and whether the event is determinant or not may be factors in determining the best course of action and associated documentation. The corrective action to be taken for analytical QC failures is usually described in the specific analytical method but may also be determined by either the supervisor, Quality Assurance Officer, or by both in conference, if necessary. Documentation is usually using the QC Outlier Report. Broader issues are more frequently addressed using the issues (corrective action) database.

3.3.1.1 Some items may not necessitate direct intervention of QA where standard practices are in place for some events, where the SOP or project or program QAP itself dictates the corrective action and where the action taken is the most conservative response practical. These types of events may be considered to have automatic QA approval.

3.3.2 A corrective action can be as extensive as replacing a complete lot of contaminated extraction solvent, re-extracting and re-analyzing a complete batch of samples, due to reagent blank contamination; or as simple as recalculating a series of results because a wrong dilution factor was applied. Again, the appropriate corrective action must be determined on a case by case basis.

3.3.3 Data cannot be released until the system is in control or the QC failure can be attributed to a cause other than method performance. In the case where the out-of-control event is due to matrix problems in the sample and the system remained out of control, the data is flagged, narrative comments usually made in the report, and supporting documentation is released to the client if needed.

3.3.4 Corrective actions are considered adequate when the problem has been resolved and data can be reported or other actions taken from an in-control condition. Alternatively, it may be determined that the action taken was, as a minimum, all that was required by the method or that no further action was reasonable or possible that would improve the data. In these cases, the final decision must be approved by the supervisor and QA.

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3.4 Documenting an Out-Of-Control Event or Corrective Action

3.4.1 This is accomplished by completing one of the following

- A Corrective Action Database (CA) Form (generated from the iLIMS issues database); see Appendix 1
- A QC Outlier Report (generated from the run sequence); see Appendix 2
- A Sample Receipt Form (for sample receipt events, see Appendix 3)
- An Audit Finding Report Form (QA use only, not shown here, see audit SOP)
- or logged onto a form which itself includes corrective actions (example, Cold Storage Logsheet, see Appendix 4).

3.4.2 CA forms are general and are for documenting corrective action taken to correct problems not specifically called out as a QC Outlier. Such events are usually more general, such as PE sample failure, questioning a general practice even if it did not necessarily precipitate an error because it could lead to problems (i.e. failing to properly document standards or misuse of a run log, etc.) These kinds of issues are documented using the CA database in the LIMS.

3.4.2.1 The CA database is searchable and can have associated documentation or link to any event requiring corrective action through references to other issues, whether CA or not. Because of this and the ability to track and follow-up on issues, it is the only acceptable mechanism to record corrective actions.

3.4.2.2 These forms may be generated for any laboratory practice, not just those technical in nature. They are free-form in the problem description and resolution with certain categories that can be assigned to individuals, links emailed to any number of people, supporting information attached, dates and priority levels assigned and parent issues linked.

3.4.2.3 These forms are accessed through the LIMS from the "Help Desk". New issues can be entered through the "Report a Problem" link and reported issues viewed under the "View Reported Problems" link. Two views appear in Appendix 1.

3.4.2.4 At least monthly all open CA issues are reviewed by QA and the laboratory director and any appropriate assignments given. In addition open issues with assigned responsibility are reviewed to determine what progress on resolution has been made and documented.

3.4.2.5 During this review the priority and status of each open issue may be changed.

3.4.2.6 As each open CA issue is resolved its status is set to 'Fixed'.

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3.4.3 QC Outlier Reports are generated by the LIMS and filled out by **technical** laboratory staff only and are designed for documenting analytical QC failures and associated corrective actions. Any failures of QC and the decision to perform corrective action or continue data processing must be documented on the QC Outlier form.

**Note: It is not necessary for analytical staff to document actions which were taken prior to processing samples or which do not in any way affect reported data.**

3.4.4 Audit Finding Reports are responded to by the assigned individual or an appropriate designee and signed off by QA or a designated individual (see the audit SOP).

3.4.5 All QC Outlier Reports and Corrective Action Forms shall be filled in completely by the person observing the event. Actions taken may be filled in by either the initiating person or the person actually performing the corrective action. The descriptions of the event and any corrective actions taken should be detailed and specific. The QC Outlier report form identifies most outlying analytical events.

**Note: Holding time violations due to laboratory error are annotated on the QC Outlier report form but can also be addressed using the corrective action report database. Holding time violations occurring due to receipt of samples beyond the criteria are documented on the sample receipt form only.**

3.4.6 If the corrective action taken and annotated on the QC Outlier Report resolves the problem and subsequent data is in control, the action is complete and only needs to be signed by the individual(s) initiating and taking action (could be either the same or different one people).

3.4.7 If the corrective action taken and annotated on the QC Outlier Report does not resolve the event and it is determined that no further action can or will be taken, the form must be signed by the analyst, supervisor, and QA.

3.4.8 Originals of all QC Outlier Reports must be included in each run sequence and associated with the data review checklist used to document the review of every validatable data package.

3.4.9 Any corrective actions taken which could either impact data directly, help to explain analytical decisions that were made in order to resolve analytical discrepancies, or which would help in the interpretation of the final data package must also be narrated in the final report. QC Outlier Report forms must be turned in with the data and the supervisor

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creating the narrative comment for that area will comment on any decisions resulting from failed QC which could impact data validity or interpretation.

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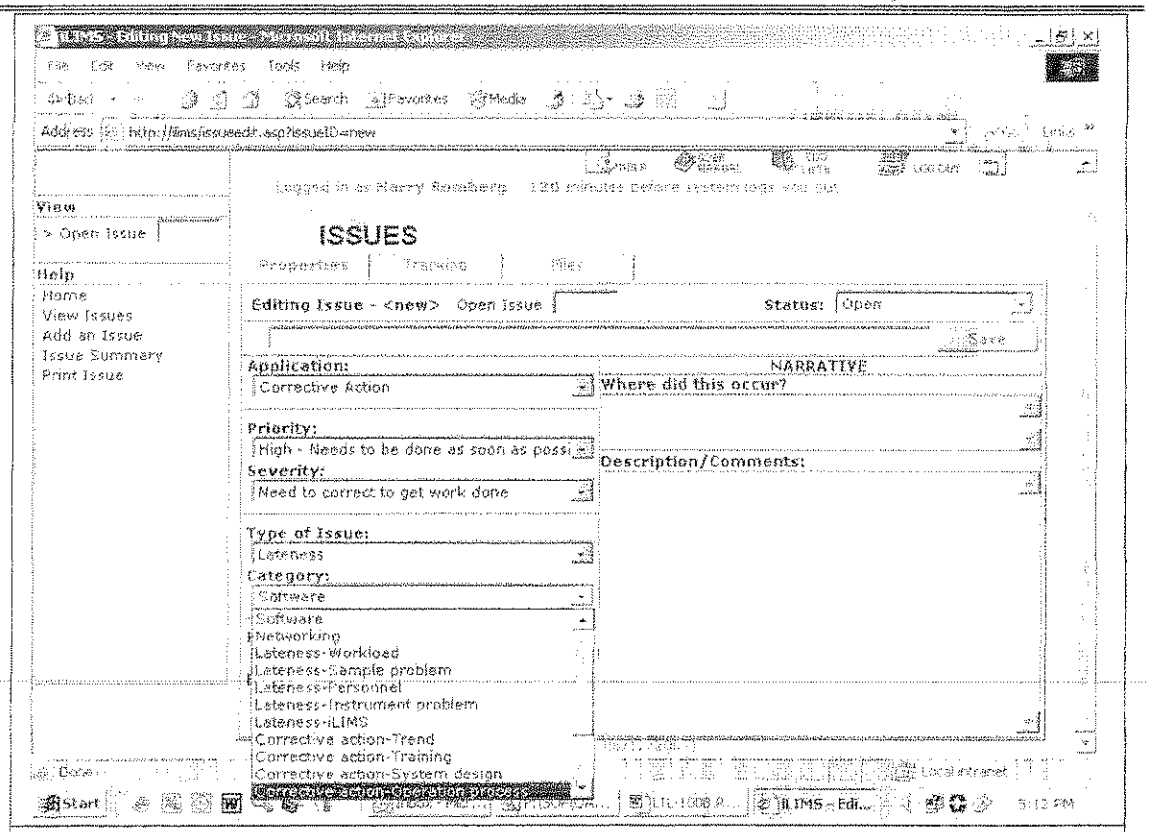
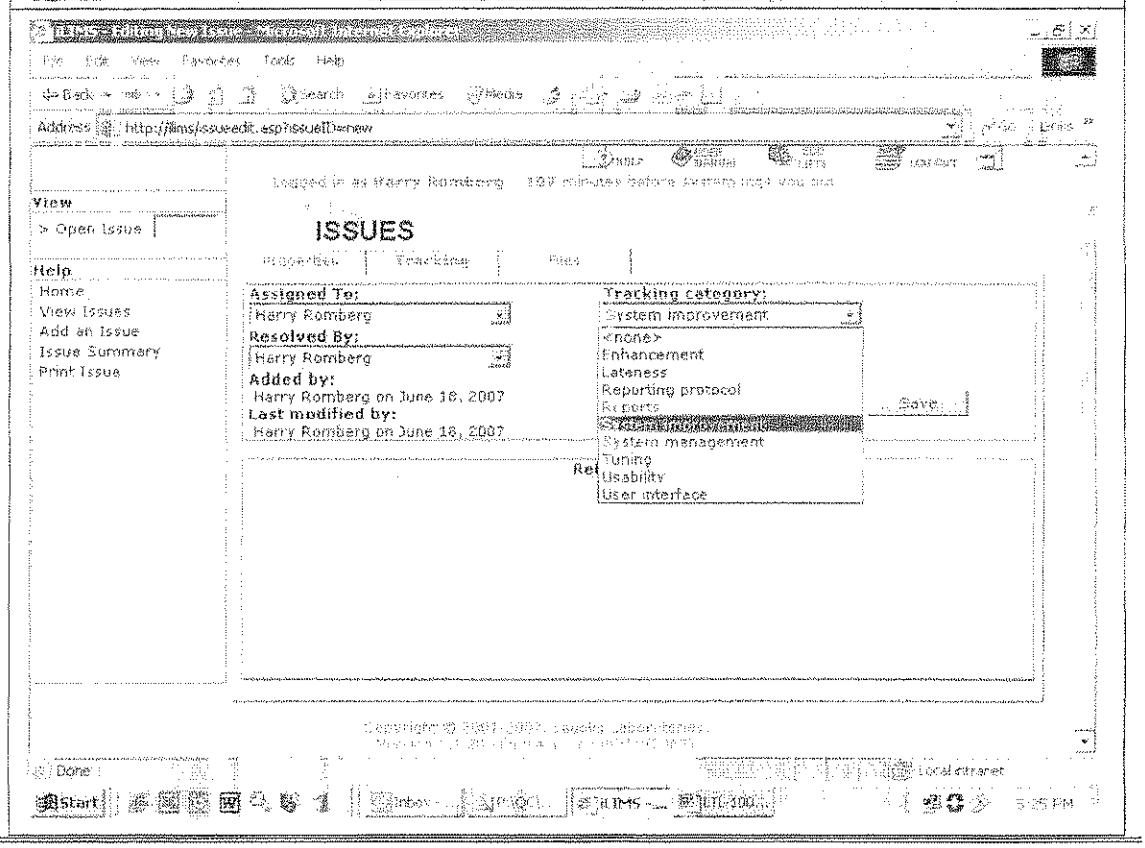
Appendix 1

Corrective Action Database Form


ISSUES
Editing Issue - <new> Open Issue
Status: Open
Application:
Corrective Action
NARRATIVE
Where did this occur?
Priority:
High - Needs to be done as soon as possi
Severity:
Need to correct to get work done
Description/Comments:
Type of Issue:
Lateness
Category:
Software
Software
Networking
Lateness-Workload
Lateness-Sample problem
Lateness-Personnel
Lateness-Instrument problem
Lateness-iLIMS
Corrective action-Trend
Corrective action-Training
Corrective action-System design


ISSUES
Assigned To:
Harry Romberg
Resolved By:
Harry Romberg
Added by:
Harry Romberg on June 18, 2007
Last modified by:
Harry Romberg on June 18, 2007
Tracking category:
System improvement
<none>
Enhancement
Lateness
Reporting protocol
Reports
System management
Tuning
Usability
User interface

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Appendix 2

QC Outlier Report

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LAUCKS TESTING LABORATORIES
RUN SEQUENCE QC OUTLIERS
FOR RUN SEQUENCE:17183

Date Current: 06/26/2007

Date Report Printed: 06/01/2007 Analyst: Colleen Conrt

Methods: 300.0

| LAB SAMPLE ID | CLIENT SAMPLE ID | ANALYTE | OUTLIER MEASUREMENT | CONTROL LIMIT | ADDITIONAL COMMENTS |
|---|---|---|---|---|---|
| ICV limits | | | | | |
| | | | | | No outliers |
| ICB limits | | | | | |
| | | | | | No outliers |
| CCV limits | | | | | |
| | | | | | No outliers |
| CCB limits | | | | | |
| | | | | | No outliers |
| Blanks | | | | | |
| | | | | | No outliers |
| BS/BSD recovery | | | | | |
| | | | | | No outliers |
| BS/BSD RPD | | | | | |
| | | | | | No outliers |
| Surrogate recovery | | | | | |
| | | | | | Not required |
| SRM limits | | | | | |
| | | | | | No outliers |
| MS/MSD recovery | | | | | |
| MG8001-005MS MS | | Chloride | 97.8% | 90 - 110 | |
| MG8001-005MS MSD | | Chloride | 98.8% | 90 - 110 | |
| MS/MSD RPD | | | | | |
| | | | | | No outliers |
| Duplicate RPD | | | | | |
| | | | | | No duplicate, not required |
| Multiple QC in QC Batch Group(s) | | | | | |
| | | | | | None observed |
| Preparation hold time exceedances | | | | | |
| | | | | | None observed |
| Analysis hold time exceedances | | | | | |
| | | | | | None observed |
| Data Inconsistency | | | | | |
| | | | | | None observed |
| Sample Name Consistency Check (Lab Sample ID Consistent with [SDG]-[Sample Number]) | | | | | |
| | | | | | None observed |

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LAUCKS TESTING LABORATORIES
RUN SEQUENCE QC OUTLIERS
FOR RUN SEQUENCE: 170.03

Date Started: 04/25/2007

Date Report Printed: 06/01/2007 Analyst: Antigen Tours

Method: 200.8

| LAB SAMPLE ID | CLIENT SAMPLE ID | ANALYTE | OUTLIER MEASUREMENT | CONTROL LIMIT | ADDITIONAL COMMENTS |
|---|---|---|---|---|---|

Additional analyzing event(s):

| Actions Taken: | | | |
|---|---|---|---|
| Check One: | | Notified: | |
| | Original results reported | | QA |
| | Rerun results reported | | Client services |

Corrective action taken by _____ on _____ Reviewed by _____ on

Description of corrective action(s): _____

Corrective actions not taken or unsuccessful (explain):

| Analyst: | Date: |
|---|---|
| Department: | Date: |
| QA: | Date: |

LTL-QCX 09

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Appendix 3

Sample Receipt Form

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Cooler Receipt Form
Laucks Testing Laboratories, Inc.

SDG: MGBG01 Taken By: CLIENT

Cooler: AAD376 Transferred: FEDEX

COC #: 1515

Project: *Mustard Gas Burial Ground (SAIC)*

Date samples were received at the laboratory: 4/11/2007

Date cooler was opened: 4/11/2007 8:30AM

A. PRELIMINARY EXAMINATION PHASE:

1. Did cooler come with a shipping slip (airbill, etc.)? YES
   If YES, record carrier name and airbill number: 859427143030
2. Were custody seals unbroken and intact at the date and time of arrival? INTACT
   Date On Custody Seal: Custody Seals Description: ONE IN FRONT AND BACK
3. Were custody papers sealed in a plastic bag and taped inside to the lid? YES
4. Did you screen samples for radioactivity using the Geiger Counter? YES
5. Were custody papers filled out properly (ink, signed, etc.)? YES
6. Did you sign custody papers in the appropriate place? YES
7. If required, was enough cooling material present? YES
8. Have designated person initial here to acknowledge receipt of cooler: _____

B. LOG-IN PHASE: Date samples were logged-in: 4/11/2007 8:35AM

Logged-in by ___ Stephen Hadley (sign) _____

9. Describe type of packing in cooler:

   BUBBLE WRAP

10. Were all bottles sealed in separate plastic bags? YES
11. Were labels in good condition? YES
12. Were all bottle labels complete (ID,date,time signature,preservative,etc.)? YES
13. Did all bottle labels agree with custody papers? YES
14. Were correct containers used for the tests indicated? YES
15. Were the correct pHs observed? YES
16. Was a sufficient amount of sample sent for tests indicated? YES
17. Were bubbles absent in VOA samples? NO
18. Temperature: 1.3

DISCREPANCIES:

ONE VIALS RECEIVED WITH BUBBLES > 1/4" PLEASE SEE SUPPLEMENTAL LOG FOR SPECIFICS.

Supplemental Sample Receipt Log
Laucks Testing Laboratories

SDG: MGBG01
Cooler: AAD449
Temperatures: 5.5
COC #: 1521

| Sample | Bottle # | Bottle Description | pH | Bubbles |
|---|---|---|---|---|
| MGBG01-010 | 0001 | 40 ml OTWS, clear glass, HCl | N/C | None |
| MGBG01-011 | 0001 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, HCl | N/C | < 1/4 |
| | 0004 | 40 ml OTWS, clear glass, HCl | N/C | < 1/4 |
| | 0005 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0006 | 500 ml cylinder, poly | 7 | None |
| | 0007 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0008 | 500 ml cylinder, poly, ZnOAc + NaOH | >12 | None |
| MGBG01-012 | 0001 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0004 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0005 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0006 | 500 ml cylinder, poly | 7 | None |
| | 0007 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0008 | 500 ml cylinder, poly, ZnOAc + NaOH | >12 | None |
| MGBG01-013 | 0001 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0004 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0005 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0006 | 500 ml cylinder, poly | 7 | None |
| | 0007 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0008 | 500 ml cylinder, poly, ZnOAc + NaOH | >12 | None |
| MGBG01-014 | 0001 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0004 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0005 | 40 ml OTWS, clear glass, HCl | N/C | None |
| | 0006 | 500 ml cylinder, poly | 7 | None |

Allowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

| | |
|---|---|
| Temperature | Allowable temperature range is 4+/- 2 degrees Celsius |
| Acid Preserved pH | pH must be less than 2 |
| Base Preserved pH | pH must be greater than 12 |
| N/C | No Checked for pH |

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Appendix 4

Example Logsheet (Cold Storage)

# Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: 2007 Correction Factor *(add this number when recording the thermometer readings)*:_____°C

| Day | Month: Time | Temp. | Initials | Actions | Month: Time | Temp. | Initials | Actions | Month: Time | Temp. | Initials | Actions |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 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 | | | | | | | | | | | | |
| 26 | | | | | | | | | | | | |
| 27 | | | | | | | | | | | | |
| 28 | | | | | | | | | | | | |
| 29 | | | | | | | | | | | | |
| 30 | | | | | | | | | | | | |
| 31 | | | | | | | | | | | | |

Record Time and Temperature in the proper blocks and initial the entry each day of normal laboratory operation.

Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/20/04

Corrective actions include:
1) Adjust the temperature of the thermostat
2) Defrost the refrigerator or freezer
3) Contact the appropriate laboratory maintenance personnel, the department supervisor, and/or the QA Officer
4) One of the above may decide that professional maintenance is necessary or even that the cold storage unit must be disposed of

Control Limits

| | |
|---|---|
| Refrigerators | 4°C ± 2°C |
| Freezers | -18°C to -20°C |
| BOD Incubator | 20°C ± 1°C |

Any and all actions MUST be recorded on this log sheet. If there is insufficient space, mark on the back of the page with the date the action occurred.
Samples MUST NOT be stored in units which are not maintaining the proper temperature.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike / PT Sample Evaluation Program**

Revision history:

| Number | Date |
|---|---|
| LTL-0048 Rev1.1 | 5/18/92 |
| 2 | 6/21/96 |
| 3 | 6/19/00 |
| 4 | 3/10/04 |

Written by: Harry Romberg Date: 3-10-04
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 3-11-04
Kathy Kreps, Laboratory Director

Reviewed
Does NOT require revisions ☒
Initials ATR Date: 6/21/05

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## 1. Introduction and Scope

1.1 Description

1.1.1 This SOP provides a description of how blind spikes are generated, what types of analyses are monitored, how results are evaluated and how Laucks handles out of specification events.

1.1.2 Materials may be from a multitude of sources. The analyst will most often be aware that the sample is a blind spike but in no case should the analyst know the "true" value of the submitted sample. On rare occasion, at the discretion of QA, a double blind sample may be submitted (one which the analyst does not know is an evaluation sample).

1.1.3 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Definition of Terms

1.2.1 **Blind Spike** - A proficiency sample which may or may not be known as such by the analyst but which contains a target analyte at a level which is not known to the analyst.

1.2.2 **Double-Blind Spike** – A proficiency sample which is submitted to the analyst in such a way that it is thought to be a routine sample and which contains an amount of target analyte unknown to the analyst.

1.2.3 **PE or PT Sample** – Performance Evaluation or Proficiency Testing Sample – Two terms with similar meaning. A vendor-supplied sample with an amount of target analyte(s) unknown to the laboratory. Typically, these are analyzed as part of a round robin study, the results of which are statistically evaluated in order to determine the range of values expected in order to meet a given confidence level. If intended strictly to test for analyst rather than lab proficiency, the sample may be purchased or prepared as a known concentration to QA and/or select supervisory staff but must not be known to the analyst.

## 2. Equipment List and Standards

2.1 Equipment

2.1.1 Pipets, flasks, containers etc. necessary to prepare spikes for submission.

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2.2 Reagents

2.2.1 Deionized water, methylene chloride, hexane, methanol and other solvents or preservatives that may be required to prepare spikes. Some samples may be prepared by outside sources as whole-volume samples which only need to be submitted to the analyst.

## 3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances. During the preparation of blind spikes, the analyst will be exposed to a variety of reagent chemicals and solvents. In addition, preservatives contained in both reference materials and in sample bottles may pose both short and long-term health hazards. The known health effects of these various chemicals may be ascertained by reading the appropriate material safety data sheets (MSDS) but the analyst should never assume all of the possible health effects are known. It is incumbent on the analyst to exercise due care and caution while executing this SOP. The company will provide any protective equipment or clothing needed to assure employee safety.

3.1.2 Many solvents also pose a fire hazard and should be treated with proper precaution.

3.2 Waste Disposal

3.2.1 Waste solvents are disposed in the appropriate waste solvent container.

3.2.2 No more blind spike material is used than is necessary for submittal of the sample so that it will not present a disposal hazard.

3.2.3 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on hazardous waste disposal.

## 4. Materials

4.1 Sources

4.1.1 Most commonly, materials are WS (water supply), WP (water pollution), soils or other materials from an external performance evaluation study. Although these are not generated directly by the laboratory, they are blind samples in that the expected values and in many cases the constituents themselves are not known to the analyst beforehand.

4.1.2 Standard materials may be purchased from a vendor, such as Environmental Resource Associates (ERA), Analytical Products Group (APG), Resource Technology Corporation (RTC), SPEX, Restek, Supelco or any other reputable vendor. If possible, Laucks uses

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NELAP approved vendors, particularly where the monitored parameters are on the NELAP PT parameter list.

4.1.3 Materials may be purchased either as Proficiency Testing (PT) samples (values unknown to the laboratory), reference materials (values known to the laboratory), or as standard materials (values known to the laboratory). They may also be made up by supervisory or QA staff from materials of known content. In any instance, the value of the components will be unknown to the analyst performing the analysis until completion of the evaluation.

4.2 Storage

4.2.1 Materials are stored as recommended by the manufacturer, most often at a temperature of 4°C ± 2°C. Metals will generally be stored in dilute nitric acid and need not be refrigerated.

## 5. Operation procedures

5.1 Requirements and Scheduling

5.1.1 These requirements may be program and/or method-specific. Laucks specific training requirements and documentation are discussed in other SOPs, department training programs and in the QA Plan. This SOP is intended primarily to document the practices and evaluation of results and not to dictate the specific analyst training and capability demonstration requirements.

5.1.2 Initially, as part of being considered able to independently perform an analysis, an analyst may be required to analyze a single blind Proficiency Testing (PT) sample. Such analyses may also just be used to supplement other capability demonstrations but not specifically required to be considered proficient. The analyst must process the samples independently, without direction or assistance in order to be considered proficient.

5.1.3 On an ongoing basis, at least annually, an analyst may also be required to demonstrate continuing proficiency by analyzing a single blind PT sample.

5.1.4 PT results may also be used as a supplement to a method verification process in order to verify the laboratory's ability to perform a method.

5.1.5 These PT samples are usually from a performance evaluation study, such as a Water Pollution (WP) or Water Supply (WS) study, an independent vendor PT, such as Environmental Resource Associates (ERA) Analytical Products Group (APG), or Resource Technology Corporation (RTC) or it may be prepared by an area supervisor from a known material. Most blind PT samples are prepared as aqueous solutions although soil PTs are periodically analyzed as well in order to meet NELAP or other

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requirements. Other ERA, APG, RTC or independent sources of materials will be used where components are not present in WP, WS, DMRQA or other "official" PT samples.

5.1.6 WP and WS program samples are currently analyzed semiannually and DMRQA samples approximately annually. It has become common to combine DMRQA analyses with the WP program, however, so independent DMRQA-only analyses are no longer commonly used by Laucks. Supplementary PT samples for analytes not present in these samples (such as fuels or GC/MS semivolatiles) are generally obtained from the same source (APG, ERA, RTC or a similar vendor). The schedule for submittal of all PT studies is at the discretion of QA in order to meet laboratory needs to qualify analysts or methods or to meet other requirements.

5.1.7 One set of PT samples may be used to qualify several analytical staff. For instance, one person may extract a sample and be so qualified. Several analysts may process the extract independently and also be qualified. If multiple analysts do process the extract, however, there must be no collaboration between analysts until the results have been received by QA.

5.1.8 In any instance, the values of the components must not be divulged to the analyst(s) prior to analysis. Furthermore, if a PT sample contains one or more components from a multi-component analysis (such as a semivolatiles or pesticide mixture), the constituents themselves must not be divulged.

5.1.9 Blind spikes should be analyzed at least in quadruplicate if used for analyst qualification, so that reproducibility can be determined as well as recovery. All results should be reported for each determination where the analysis was otherwise in control. Evaluation of replicates is a laboratory option and is rarely required of any external proficiency testing program.

5.1.10 Blind spikes or PT samples are typically analyzed for the following analyses (generally in water and in more limited cases in soil):

- ICP metals
- ICP/MS metals
- Mercury
- GC Volatiles (BTEX & MTBE)
- Diesel
- Hexane Extractable Material (1664 oil & grease, water)
- Pesticides
- Herbicides
- GC/MS Volatiles
- GC/MS Semivolatiles (including PNAs)

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- Cyanide
- Total Organic Halogens (water)
- Total Organic Carbon (water)
- Phenolics (water)
- Ion Chromatography (F, Cl, $NO_3$, $SO_4$)
- $NO_3/NO_2$ Automated Cd reduction (water)
- pH (water)
- solids (water)
- turbidity (water)
- others at the discretion of QA

5.1.11 Where other methods are very similar (i.e. 200.7 and 6010 for ICP metals), the same PT analysis may be considered adequate documentation for both methods. Other blind PT studies may be conducted at the discretion of QA.

5.1.12 Samples will be given a laboratory ID number and test code when they are submitted to the laboratory and should be tracked in the same manner as a routine sample. Results will be compared against vendor-supplied, method-specific, or laboratory-derived limits as noted in the Evaluation and Reporting section.

## 6. Evaluation and Reporting

6.1 Data Package Organization

6.1.1 Paperwork must be completed as it would for routine samples, documenting preparation, calibration, and analysis and quality control. In addition, a summary page must be completed with the results of the sample and any replicate analysis. The summary page must contain the following elements:

- Analyst
- Date of analysis
- Preparation Technician (where appropriate)
- Date Prepared
- Analysis (Method*)
- Preparation (Method*)
- Components obtained from the analysis
- Results obtained from the analysis
- Replicates (where applicable) and associated RPDs

* At the discretion of QA, analysis and preparation methods may be considered sufficiently similar to qualify for more than one reference technique.

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6.1.2 If the results are to be submitted to a vendor for statistical evaluation and in order to meet lab PT requirements, they should be submitted to QA on the vendor-supplied reporting forms. Although these are generally transcribed by QA into web-based reporting systems or onto final forms that are physically submitted to the vendor, this reduces the potential for an error based on QA personnel rather than the analyst which would misrepresent the actual proficiency of the analyst or analytical system.

6.2 Evaluation

6.2.1 The data will be evaluated by QA with possible assistance from other supervisory staff. Data must meet the limits supplied by the vendor, if purchased or supplied as part of a PT program. If limits are not given by the vendor, method specific limits may be adopted or the laboratory may choose to accept recoveries based on internal QC limits.

6.2.1.1 All relevant components must be identified by the analyst, although in a few limited cases, similar components react in much the same fashion (i.e. similar retention times or patterns). In these instances, at the discretion of QA, the analyst may be allowed to re-evaluate the analysis.

6.2.1.2 If the analysis is a multi-component mixture, the results may be considered acceptable if at least 90% of the target analytes are quantified correctly as long as all are at least identified. This may NOT be acceptable to programs which qualify the laboratory on an analyte-specific basis in which case a second PT sample may be required.

6.2.1.3 Replicates will most often be evaluated where recovery exceptions occur or where it is determined by QA or the area supervisor that this reproducibility is a critical part of the analyst's evaluation. They will also be evaluated if it is so specified in the reference method. In these instances, the acceptability criteria are generally either the laboratory-derived RPD(s) or the reference method-specified criteria.

6.2.1.4 At the discretion of QA, the data may also be evaluated for completeness and documentation.

6.3 Remedial Actions

6.3.1 If the limits for the analyzed material have been exceeded, that performance criterion will be considered to have not been met. In such case, the data will first be re-evaluated by the analyst. If sufficient extract/digestate remains, this may include re-analysis.

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6.3.2 If, after re-evaluation, the performance criterion still has not been met, the results from the entire analysis will be evaluated and if sufficient criteria have not been met, the analyst may be required to analyze another blind PT sample.

6.3.2.1 In some cases, the quality of the vendor-supplied material may be in question. In this instance or in the case where no more of a specific material is available in a timely fashion, a second source of performance evaluation material may be used.

6.3.3 Continued failure may result in either or both examining the analysis/preparation method for discrepancies or it may require re-training of the analyst if it is determined that the method and instrumentation is functioning properly. In either case, action must be initiated immediately to insure that accurate results are being produced for actual laboratory samples.

6.3.4 In the extreme case, it may be determined after consultation with supervisory staff and laboratory management (including QA), that no analyses can be performed using that method or that analyst until there is demonstration of adequate proficiency.

## 7. Record Keeping

7.1 Analyst and Method

7.1.1 Records for all evaluations will be maintained by QA. Analyst evaluation will be maintained in the analyst's training file or in method-specific files or both.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1011

Title: **Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits**

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## 1. Introduction and Scope

1.1 Overview

1.1.1 This SOP describes the determination of Instrument Detection Limits (IDLs), Method Detection Limits (MDLs), Precision and Accuracy (P&A) studies, the setting of Reporting Limits and the determination and use of control limits, including a discussion of laboratory uncertainty. Although P&A studies may be used as part of an analyst's Initial Demonstration of Capability, that is discussed in the Documentation of Analyst Capability and Training SOP (LTL-1004) and not here.

1.1.2 In general, detection limits are the minimum amount of a target analyte that can be measured and determined to be greater than zero with a known degree of confidence. For purposes of this SOP, the known degree of confidence for MDLs will be defined as the 99% level. IDLs are based strictly on instrument response and MDLs on a sample processed through the entire preparation process. This SOP is based on information provided in 40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11* and in other sources such as the EPA Contract Laboratory Program (CLP) Organic and Inorganic Statements of Work (SOWs), SW846, the latest relevant National Environmental Laboratory Program standard and the latest Department of Defense (DoD) Quality Systems Manual (QSM) for Environmental Laboratories.

1.1.3 According to EHSG MICE methods support hotline for SW 846, at the present time, the EPA recognizes that the existing statistical methods of determining MDLs often lead to low values that are difficult to support and can lead to the reporting of false positive values as well as unreliable negatives at levels as low as the MDL would suggest. Different agencies may choose to handle this in different ways, some relying on a low level check standard as the threshold. However, at the present time, MDLs are still determined per the 40 CFR reference for most clients and applications. Often times, if there is no change in instrumentation or methodology, once the MDL is determined, it only needs verification on a periodic basis. Laucks uses these verification data not only to verify detectability but to demonstrate that the MDL remains valid and has not changed significantly or to justify resetting the MDL if it HAS changed. This SOP does not attempt to address all of the agency or client variability, only how MDLs are determined and documented when and if they are required. Since they are still required by most programs in which Laucks participates, they are still in general use by the laboratory until such time as there is broad acceptance of an alternative. Laucks will, however, continue to abide by the requirements of clients and agencies under whose auspices we do business.

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1.1.4 Criteria for Precision and Accuracy (P&A) Studies are often defined in the specific published method, particularly those in SW 846. Where criteria are not so defined, Laucks has chosen to either use the criteria from similar methods or to set in-house criteria based on the judgment of senior management and QA, often generated from in-house statistics. Where two methods are the same in technical detail and one does not provide P&A criteria, performance under the guidance of the method with specifications may be used to satisfy the performance criteria of both.

1.1.5 Control limits for a method are initially determined using limits supplied in the method or defined by the program (such as CLP, DoD or AFCEE). After sufficient points have been accumulated the laboratory performs a statistical analysis of the data and computes the control limits which are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation. Additionally, some accrediting authorities/agencies may define limits that are not otherwise set in the reference methods. Laucks, at our discretion, may decide to use these either just for that agency or for all cases where they are not otherwise defined.

1.1.5.1 Control charting is generally done using the laboratory-generated statistical control limits. Control charting is not a topic of this SOP but is thoroughly addressed in the QA Plan, Section 7.6.

1.1.6 This SOP is designed for applicability to a wide variety of sample types ranging from reagent water to solids containing the target analyte. The MDL may vary as a function of sample type. Laucks rarely determines MDLs on any matrix other than soil or water but would do so if there was a sufficient sample load anticipated and if the client requested such an effort. Other MDLs may be estimated based on these studies.

1.1.7 This SOP requires that a specific, detailed analytical method exist. When determining MDLs and P&As following this SOP, it is imperative that all sample processing steps included in the analytical method be included.

1.1.8 Where a specific method has requirements exceeding the requirements of this SOP, that method will take precedence. Where a reference method has stated detection limits, these are generally taken to be estimated MDLs which are dependent on the exact methodology, instrumentation and performance characteristics of the system used while developing the method. Although Laucks would strive to meet or exceed method specified limits this may not always prove to be possible. This SOP is to be followed to validate a new method or to validate a significant change in a current method.

1.1.9 MDLs are to be determined approximately annually or as needed for procedures that are performed on an infrequent basis. For common procedures MDL check standards will be

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used. The laboratory may statistically analyze a recent series of MDL check standards in order to update MDLs or to determine if updating is required.

1.1.10 PCB and multi-component pesticide MDLs are to be performed for each PCB/pesticide to be analyzed. However, due to the inability to analyze all of these multi-component species in one solution, the work to do all of these analytes annually is considered impractical. For PCBs, Aroclor 1660 (1016/1260) is verified monthly and the other Aroclors in sequential order each month until they have all been verified and then the process starts over. The verifications, as with single component analytes, are used to periodically assess the MDL.

1.1.11 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis except in the case of P&A studies which are used to demonstrate the capability of the analyst.

1.2 Method Description

1.2.1 Detection Limits

1.2.1.1 For some metals methods (i.e. ILM04.2 and DOD QSM, ver. 3), the Instrument Detection Limit (IDL) must first be determined. The IDL may also be determined strictly for informational purposes for other methods but is not required. The IDL allows the analyst to assess the precision of the measurement system and to estimate the target concentration for the MDL study. IDLs generally follow the ILM04.2 and earlier model and are determined by analyzing 7 low-level standard replicates on 3 non-consecutive days and averaging the sample standard deviations from each of the three days. In this model, the IDLs are generally required to be performed quarterly. In newer CLP methods (i.e. ILM05.3 and above), IDLs are not required as they have gone to the MDL model.

1.2.1.2 In order to determine MDLs, a minimum of seven replicate measurements are made of a prepared sample matrix which contains approximately 1 to 5 times the estimated detection limit. A Student's t determination is made for the number of data points available, usually 7 (6 degrees of freedom), and the resulting standard deviation multiplied by that value to determine the MDL.

**Note:** The CFR states that the recommended concentration levels used to determine the MDL be one to five times the MDL. It later implies that a level of up to 10 times the MDL is acceptable. Laucks considers up to 10 times the MDL to be an appropriate concentration although limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

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1.2.1.3 In many instances, immediately after an MDL study has been performed and at any later time determined to be necessary when reporting results near the MDL, a low level check standard may be used to verify a previously established MDL. This check standard is generally run at 2-3 times the statistical MDL (up to 4x for multi-analyte methods) and must be detected to be considered as verifying the MDL. It must be analyzed on every instrument used for reporting data and the only requirement Laucks imposes is that it be detected at a minimum of 3 times the background noise level.

1.2.1.4 The laboratory will more often run a check standard to verify the Reporting Limit (RL), a value usually significantly above the MDL. The RL is generally at a level where identification and quantitation of the target analyte is more reliable and below which any values should be considered estimated at best.

1.2.1.5 Reporting Limits (RLs) are set by the laboratory as limits that can be reliably reported on a consistent basis with a reasonable degree of confidence that the reported level is accurate. These limits may be set at the Practical Quantitation Limit (PQL) initially by using a multiplier times the MDL. The multiplier is rarely defined in the method but Laucks will commonly use at least 3 to 5 times the MDL or even higher. After initial setting of the RL, it is rarely changed unless significant changes in the MDL occur which make it necessary to raise or lower the RL.

1.2.2 RLs can also be agency or project driven. As long as they are approximately equal to or greater than the laboratory established RL, they can be considered reliable. Values significantly below the laboratory-established RL, however, will be less reliable and sample values in this range may be considered estimated.

1.2.3 Precision and Accuracy (P&A) Studies are studies performed in order to demonstrate the laboratory's ability to perform a method and are also used to demonstrate analyst capability to perform the method. They generally involve the analysis of 4 replicates spiked at concentrations defined in the method but generally near the mid-point of the calibration curve or 10 to 50 times the MDL for each analyte. Adequate performance is sometimes defined in the reference method, although if the method performance has been demonstrated, analyst capability may be demonstrated in comparison to laboratory limits.

1.2.4 Control limits may be specified in a reference method, in federal agency guidance documents or may be statistically determined by the laboratory from existing data. In general, laboratory determined limits for control samples should not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory cannot demonstrate adequate performance for all target analytes on a routine basis. However, if

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the exceptions are limited to a few analytes and they are not vastly different from the program limits, this may not be an indication of poor performance as much as it is a reflection on the performance of those particular analytes.

1.2.5 Control limits may be specified in a reference method, may be statistically determined by the laboratory from existing data, or may be administratively set in project QAP or federal agency guidance documents. It is desirable that laboratory determined limits for control samples not exceed specified limits. However, for multi-component determinations that include analytes that are known to be poor performers, it may not be feasible to use non-statistically derived control limits. This will be observed most frequently for organic methods. As a consequence, the default 70-130% limits listed in SW 846 Method 8000, for instance, are frequently unreasonably narrow, as evidenced by many documents including the Department of Defense (DOD) Quality Systems Manual (QSM) and the AFCEE QAPP, all of which have statistical bases and are substantially wider than those in Method 8000. If laboratory determined limits do exceed method-specified limits, the system must be evaluated. Such an evaluation will include an assessment of the actual compound and comparison to control limit data from other sources (such as the DOD QSM). This evaluation should be documented on the spreadsheet or other paperwork associated with the control limit determination and any exceptions or special considerations discussed. It should be noted here that, for some compounds, no laboratory can consistently achieve the default method criteria.

1.2.6 That said, in most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory's statistics would indicate that it cannot demonstrate consistent performance within the specified guidelines for all samples on a routine basis and that a QC sample that was within the specified limits would not necessarily indicate that the other related samples would also be within specifications. However, if the laboratory is clear about the internal limits that are being used, they are statistically based (rather than default values) and the user finds them acceptable, the laboratory may simply identify when the project or method limits are being exceeded and if they meet the internally calculated limits, the system will be considered as meeting the data quality objectives.

1.2.7 It is not uncommon for clients to specify reporting or control limits in their project quality assurance plans. As long as they are achievable (i.e. the requested RL is not lower than the laboratory MDL), Laucks will generally comply with the client's request for that particular project, although frequently flagging will be used to indicate values are estimated below the RL. A low-level standard may sometimes be used to show that a specific RL which is below the laboratory-established level is achievable but care must be taken in these instances. Quantitation will still be unreliable and if the statistics show it does not fall within the 99% confidence interval for MDLs, even if the standard is

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detectable, values in that range may still be falsely positive or negative and estimated. In some cases, pattern or spectrum matching may help to confirm the presence of an analyte at very low levels but this still would not improve the reliability of the actual quantitation.

1.3 Definition of Terms

1.3.1 **Accuracy** - The degree of agreement of a measurement (or an average of measurements of the same thing), X, with an accepted reference or "true" value, T, usually expressed as the difference between the two values, X-T, or the difference as a percentage of the reference or true value, 100*(X-T)/T, and sometimes expressed as a ratio, X/T. Accuracy is a measure of the bias in the system. Accuracy shall be calculated as follows:

$$\%R = \frac{C_s - C_u}{S} * 100$$

Where:

Cs = Concentration of spiked sample
Cu = Concentration of unspiked sample
S = Expected concentration of spike in sample
%R = Percent recovery

1.3.2 **Control Limits** - Control limits may be specified in a reference method (either as mandatory or guidance limits), or may be developed by the laboratory using internal performance data. Control limits represent acceptance criteria for determining whether an analytical system is in control (functioning within acceptable guidelines).

1.3.3 **Control Sample** - A QC sample introduced into the analytical process to allow evaluation of the measurement system. In general, it is best to use control samples of a matrix similar to the samples being analyzed, where such are available. The control sample, however, will generally be free from interferences other than those inherent to the matrix itself.

1.3.4 **Degrees of Freedom** - The number of independent estimates that could be obtained from a specific set of data. In general, for a simple set of *n* independent values,

$$df = n-1$$

1.3.5 **IDL** - Instrument detection limit - The lowest concentration of a target analyte that can be measured and known to be greater than the instrumental background with a known degree of confidence. It may be used as a starting point for selecting MDL study spiking levels.

1.3.6 **MDL** - Method detection limit - The minimum concentration of a substance that can be measured and reported with a known degree of confidence (99% for our purposes) that

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the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

1.3.7 **Mean** - The arithmetic sum of a set of observations divided by the number of observations.

$$\frac{\sum X_i}{n}$$

Where:

$X_i$ = sample value for replicate $i$
$n$ is the number of replicates

1.3.8 **P & A** - Precision and Accuracy - This often refers to a study conducted to validate a method or an analyst conducting a particular method. It is a measure of the degree of reproducibility of a series of measurements and the degree with which those measurements match the true value.

1.3.9 **PQL** - Practical Quantitation Limit - The limit at which it is determined that the constituent can not only be detected but be accurately quantified. This limit is usually 3 to 10 times the MDL but may be even larger depending upon the constituent and the matrix. Factors are often taken from the published method but may be set by the laboratory if published factors do not exist. These limits may also be used as the routine reporting limit (RL), unless otherwise contractually defined.

1.3.10 **Precision** - Reproducibility. A measure of mutual agreement between individual measurements of the same property, usually under prescribed similar conditions. Precision is best expressed in terms of the standard deviation. Various measures of precision exist depending upon the "prescribed similar conditions".

1.3.11 **Reporting Limit (RL)** - A value greater than or equal to the MDL or the IDL which may be based on QA decision, the published method specifications, or project-specific requirements. The RL is the value the laboratory considers to be both qualitatively and quantitatively supportable and the lowest at which results are routinely reported without being flagged estimated or without the express understanding of their estimated nature.

1.3.12 **Standard deviation** - A statistical measure of the variability of a set of sample observations. For the purposes of this SOP, the sample standard deviation is used. This is calculated using the formula:

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$$s = \sqrt{\frac{\sum\left(X_i - \overline{X}\right)^2}{n-1}}$$

Where:

s = the standard deviation estimated with n-1 degrees of freedom.
$X_i$ = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
*n* = the number of replicates

## 2. Equipment List and Standards

2.1 Equipment, Reagents and Standards

2.1.1 As appropriate for the given analysis.

2.1.2 Personal Computer with access to a spreadsheet program such as *Microsoft Excel* and the laboratory MDL database.

## 3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Refer to the specific analytical SOP for appropriate safety precautions.

3.2 Waste Disposal

3.2.1 Refer to the specific analytical SOP for appropriate waste disposal practices. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

## 4. Calibration and Quality Control

4.1.1 Calibration is as appropriate to the specific method. No matrix spiking or other routine QC is required beyond the actual preparation of the IDL, MDL or P&A studies.

## 5. Responsibilities

5.1 Analyst

5.1.1 Each analyst is responsible for verifying a valid MDL study was performed and is available for each method/matrix they perform except in instances where an actual study is not practical or meaningful (i.e., pH or conductivity). In addition, where there are multiple instruments (primarily the organic department and ICP/MS), the analyst is

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responsible for verifying a valid annual MDL was performed on each instrument for each method/matrix they analyze. In those instances where an MDL is **not** required by a method or agency (see 1.1.3), this requirement may be waived as long as the analyst appropriately verifies the MDL in whatever way **is** required. **Note:** When full MDL studies are required, they can be evaluated using the 'MDL Calculation Workbook' report available in the LIMS. When monthly MDL verification standards are used to determine the MDL, this may be done by LIMS managers or QA.

5.1.2 Each analyst is responsible for producing a personal one-time initial demonstration of precision and accuracy. This is usually done on a water matrix.

5.1.3 A metals analyst is responsible for assuring that a quarterly IDL study is produced on each instrument.

5.1.4 Each analyst is responsible for labeling MDL and P&A studies appropriately.

5.1.5 Each analyst is also responsible for analyzing and reporting the MDL verification check samples each month for all method/matrix combinations he/she is responsible for AND when normal samples for the same method/matrix are logged in during the month or however frequently they are needed.

5.1.6 Each analyst is responsible for turning in a legible MDL, IDL (where required), and P&A study to their supervisor for review and approval prior to final submittal to QA.

5.1.7 All of the analyst activities should be coordinated through the area supervisor.

5.2 Department Manager or Senior Analyst

5.2.1 Each department manager, supervisor or senior analyst is responsible for coordinating the effective completion of the required studies and verification standard analyses. This may include but not necessarily be limited to helping determine appropriate concentration levels, coordinating the completion of the study within the timeline required by the method and/or the QA department, and scheduling the study around the analytical workload. If the supervisor or senior analyst also conducts analyses, they must also make sure they have demonstrated capability as well as other staff.

5.2.2 It is the responsibility of the area supervisor or senior analyst to insure that the analyst is performing the studies and verifications within the guidelines of the method and to perform a review of the final data prior to submission. This review should include determination that appropriate spiking levels were used, that the data was properly computed and transcribed, and that any problems or concerns encountered during the study are documented. Part of this review must include the comparison of the data to method specific criteria. In other words, P&A data must be compared to established

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method criteria and MDLs must be compared to Reporting Limits to ensure they are no greater than the RLs.

5.2.3 It is the responsibility of the area supervisor to obtain the necessary information to update the control limits at a minimum of annually or to assist in verifying that updates are not necessary because the MDL hasn't changed significantly. This may be done in conjunction with QA and the LIMS/MIS department.

5.2.4 The department manager, in conjunction with QA, will also be responsible for maintaining files for all methods and staff qualified in their department.

5.3 QA Department

5.3.1 It is the responsibility of the QA department to issue a Corrective Action notice to any department who fails to turn in acceptable MDL, IDL, or P&A studies or verifications.

5.3.2 It is the responsibility of the QA department to work with supervisors to schedule studies and to maintain files of all current and historical studies.

5.3.3 QA will review and provide the final sign-off that the study meets requirements.

5.3.4 QA will review and provide the final sign-off of reporting limits.

5.3.5 QA will bear the responsibility to maintain the statistically determined control limits and to ensure that they are within those specified in the reference method. QA will require assistance from the LIMS management.

## 6. Operation procedures

6.1 General

6.1.1 All studies must be assigned to projects and given laboratory LIMS SDG numbers. If done properly, this allows us to electronically track and retrieve any needed data. Hardcopy studies may be kept by the individual department as demonstrations of method or analyst capability. Projects will almost always be assigned by QA, though pricebooks (test lists) and SDGs can be assigned by many supervisory personnel. Most studies will use the LIMS client code QA Officer in order to better track them at a later date.

6.2 Instrumental Detection Limits (IDLs)

6.2.1 It is rarely necessary to perform actual IDL studies except for metals analyses. For metals analyses, they are performed quarterly on each instrument (for ILM04.2 and DOD QSM ver. 3 for SW 846 6010 & 6020 methods). IDLs are not required for ILM05.3 or later CLP SOWs. Studies may be useful, however, to demonstrate instrument capabilities and

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as a tool for estimating the Method Detection Limit (MDL). Although IDLs may be used as estimates to determine appropriate MDL spiking levels, it is strictly prohibited to compute the actual MDLs based on IDL determinations. The following guidelines are provided for several general class of analyses, regardless of whether an IDL is required for that analysis type.

6.2.2 As with all studies, a laboratory ID number should be assigned by QA for tracking purposes. In the case of metals IDLs, the same ID number may be assigned to all of the quarterly IDLs, rather than just one per instrument.

6.2.3 Actual IDLs studies are performed according to the CLP SOW by analyzing 7 replicates of low-level standards made up in the same matrix as all standards and not including any processing steps that would not ordinarily be performed on standards. The levels of those standards should be estimated from manufacturers detection limit specifications or from past experience.

6.2.4 IDLs should be performed under the same instrumental conditions as will be used to perform actual analyses.

6.2.5 IDL studies must contain the following information (not necessarily in this order) for submittal to QA.

- Laboratory ID number
- Analyst who performed the IDL study
- Instrument name and ID which will distinctly identify that instrument
- Spike level
- Measured concentration of the 7 replicates (per day)
- Standard Deviation
- Mean
- Determined IDL
- Concentration Units
- Date(s) the study was analyzed
- Analysis (i.e. ICP, ICP/MS, etc.)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.2.6 Spectrophotometry

6.2.6.1 The ILM04.2 EPA/CLP SOW for metals requires that the IDL study be run on 3 non-consecutive days at least 7 times each day. It is prepared from an acidified aqueous standard solution made up at 3 to 5 times the manufacturers suggested IDL. The sample standard deviation (n-1) for each individual set of determinations is calculated and the

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final IDL is calculated as 3 times the average of the standard deviations for the three days. This may be performed using any commercial spreadsheet but care must be taken to insure that it is done using the sample standard deviation (n-1) calculation. For *Microsoft Excel*, this is the =STDEV() calculation. Ten percent of the calculations must be manually verified in order to demonstrate that the spreadsheet calculations are accurate. The newer ILM05.3 CLP SOW does not require IDL studies.

6.2.6.2 If other spectrophotometric method IDLs are established by analyzing standards 7 times on 3 non-consecutive days, the calculation of the IDL is performed as described above.

6.2.6.3 Versions of the EPA/CLP methods prior to the Inorganics Statement of Work (SOW) ILM05.3 do not prescribe the determination of MDLs. It is standard laboratory procedure to perform an MDL study (see section 6.3) approximately annually for almost all routine methods of analysis, regardless of IDL frequency or other determinations.

6.2.7 Chromatography

6.2.7.1 The analyst should estimate the signal:noise for determining concentrations to use for an IDL study. A preliminary estimate of 5x signal:noise is to be used; if necessary this will be adjusted and the study repeated.

> **Note:** Neither Gas Chromatography, HPLC or GC Mass Spectrophotometric (GC/MS) analyses typically require the analysis of IDLs. However, if circumstances or requirements dictate, the above and below paragraphs may be considered in their determination.

6.2.8 Gas Chromatography/Mass Spectrophotometry

6.2.8.1 Mass spectral identification criteria are key in selecting target concentrations for the IDL study. The mass spectroscopist's experience in determining the minimum identifiable concentration must weigh heavily in selecting concentrations. All compounds must meet the spectral matching characteristics as called out in the analytical method for the IDL study to be valid.

6.3 Method Detection Limits (MDLs)

6.3.1 As stated in the 1.1.3, the EPA and other agencies are beginning to realize the tenuous reliability of the current statistical treatment of MDLs. However, most methods still require these studies. MDL studies must be performed or verified annually for each method/matrix combination for inorganic analysis and for each method/matrix/instrument

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combination that will be used for most organic methods and ICP/MS where there are multiple instruments.

6.3.2 Method detection limits may be determined by one of two paths. The full MDL study is the first one described below. This method is used when a new method or instrument is introduced or changed in a major way in order to qualify that method / instrument for immediate use. It may also be used where Laucks performs a particular analysis irregularly so that recent cumulative MDL verification checks are not available for our more automated method. The second method is to use recent cumulative MDL checks to either determine a new MDL over the course of several months or to prove that the MDL has not changed significantly enough to require being re-established.

6.3.3 MDL Study

6.3.3.1 MDL studies must also be performed when any major changes have been made in an instrument, such as a detector change.

6.3.3.2 Prior to beginning an MDL study, a LIMS laboratory ID must be created within the QA Officer's MDL project. The data generated from the study is then referenced to that SDG in the same manner as routine sample data.

6.3.3.3 MDL studies must contain the following information (not necessarily in this order). This should be accomplished using the Excel spreadsheet available for attachment to the LIMS Run Sequence (See Appendix 2). This spreadsheet is also used to calculate and evaluate MDLs based on criteria in 40CFR part 136 and some inhouse guidelines.

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation (where applicable)
- Date(s) the study was prepared
- Method number of the clean-up (where applicable)
- Analyst who performed the MDL study
- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 7 replicates
- Standard Deviation
- Mean
- Determined MDL

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- Concentration Units
- Reporting Limits (RLs)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.3.3.4 The analyst must compare the MDLs with their current Reporting Limits (RLs) to ensure that they are no higher than the RLs. In fact, in most cases the MDLs should be no higher than 1/3 of their respective RLs unless there is a specific request to report down to the MDL.

6.3.3.5 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL and cannot be re-determined with more appropriate results), the QA Officer and the supervisor, often in concert with the Laboratory and/or Technical Director(s), must meet to determine the appropriate course of action. Reporting limits are intended to be at a level for which method precision and accuracy can be obtained. This generally cannot be done when the RL is too close to the MDL.

6.3.3.6 In order to determine the Method Detection Limit (MDL), it is first necessary to estimate what the MDL will be in order that the appropriate spiking levels may be used. How this estimate is made is immaterial to the actual MDL determination. Methods for making this determination may include any one or a combination of the following:

- estimating based on the instrument detection limit (IDL) as determined above or by any other means
- estimating based on the previous MDL
- estimating based on 3 times the instrument signal to noise ratio
- estimating based on analyst judgment

6.3.3.7 A solution is then prepared and spiked into a sample matrix, which is as free as possible of interference and target analytes, at a level that will result in a sample concentration equivalent to 1 to 5 times the estimated MDL.

**Note:** 40 CFR part 136 states that if the **measured** level of analyte (not the "true" value) be no greater than five times the MDL. We interpret this to mean that the mean of the measured values be compared to the determined MDL. It later implies that a level of up to 10 times the MDL is acceptable. The analyst should make his/her best effort to spike at a level within 5 times the measured concentration. At least 2/3 of aqueous MDLs should be within this range. However, Laucks considers up to 10 times the MDL to be a sufficient concentration for soils and the remaining aqueous analytes. Limited exceptions to this rule may be granted under special circumstances as long as the deviations are not great and they are approved by QA.

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6.3.3.8 Reagent (blank) water should be as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interference concentrations are not detected at the estimated method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species. The interference concentration is presupposed to be normally distributed in representative samples of a given matrix.

6.3.3.9 Recovered spiking levels which are determined to be less than 1x the MDL should be reanalyzed at a higher spike level. Recovered spiking levels greater than 10x the MDL should in most circumstances be re-analyzed at a lower spiking level.

6.3.3.10 On occasion, the statistically derived limits can be so low that one would not actually be able to verify the presence of that analyte and would not be able to call it a detected value. A logically derived administrative MDL may need to be set in this instance.

6.3.3.11 Likewise, for an analysis that exhibits a great deal of variability, the analyst may actually still be able to observe a peak or pattern below what the statistics suggest.

6.3.4 MDL Verification Check Alternative

6.3.4.1 The same basic concepts apply as for the full MDL study with some important distinctions. The primary difference is that the MDL data will be accumulated from monthly MDL verification checks that are assigned by QA.

6.3.4.2 Each month, QA assigns MDL check samples through the LIMS to those analyses we do frequently enough (1 per month for at least 7 months per year) to accumulate enough data over the course of a year to recalculate the MDL. Each of these data should always demonstrate that the existing reportable MDL is being met.

6.3.4.2.1 If the individual MDL check does NOT support the reportable, MDL, either the MDL needs to be re-established or the reportable MDL will need to be raised to a level we CAN support.

6.3.4.3 Approximately annually or after 7 data points have been accumulated over the course of the year, the data for all analyses so treated will be downloaded and processed to determine a new MDL value.

6.3.4.3.1 If the MDL for a particular analyte/method has NOT changed significantly from the reportable MDL established (or verified) the previous year, the data are stored and no further action is needed.

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6.3.4.3.2 If the value HAS changed, particularly if it is higher than the one determined previously, the reportable MDL in the LIMS will most likely need to be changed or other actions taken to enable the lab to achieve that MDL.

6.3.5 Preparation of Spiked Samples

6.3.5.1 The MDL is almost always determined in reagent water or clean sand. Glass beads or other materials may be used under specific circumstances. Prepare a laboratory standard containing all analytes of interest at a concentration which is at least equal to or in the same concentration range as the estimated MDL. The analyte concentration should not exceed 5x the estimated MDL but allowances may be made up to 10x the determined MDL.

6.3.5.2 It is extremely rare that Laucks will perform studies for other than reagent water or soil. Soil matrix will almost always be represented by clean blank sand except for metals analyses where even clean sand contains levels of some metals which exceed the 10x acceptance criteria. For such analyses, glass beads (preferable) or reagent spikes are used containing only the digestion/preparation reagents. MDLs on other matrices will generally only be performed upon specific client request.

6.3.6 Calculation of recovery statistics

**Note:** All values are used without correcting for native concentration. As previously mentioned, if blank correction is a part of the method, the average blank value is used for correcting analyte concentration measurements. In almost all methods, however, blank correction is forbidden.

6.3.6.1 The sample standard deviation is calculated as follows:

$$s = \sqrt{\frac{\sum\left(X_i - \overline{X}\right)^2}{n-1}}$$

where:
s is the standard deviation estimated with n-1 degrees of freedom.
$X_i$ = sample value for replicate *i*
$\overline{X}$ = mean of all of the replicates
*n* is the number of replicates

6.3.6.2 The Student's *t* statistic is determined for (*n* -1) degrees of freedom at the 99% confidence interval (CI). A Student's *t* table for the 99% CI is provided in Appendix 1. For most data sets, using n=7 sample readings, the *t* value is 3.143.

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**Note:** In some cases, it may be determined that it is useful to prepare an additional sample so that, in case of laboratory accident, at least 7 are available for statistical analysis. Whether or not this is done, all samples analyzed **must** be used in the statistical evaluation unless there is a strong reason to reject one or more of the data sets, such as obvious contamination, abnormally poor surrogate recovery, values that are in obvious and significant disagreement with all of the others, or spilled sample. It is inappropriate to reject data which do not have an overriding reason to do so. The reason for rejection must be clearly documented in the data file. If more than 10 repetitions are used in the MDL determination, the current standard MDL spreadsheet will not accommodate the calculation.

6.3.6.3 The MDL determination then becomes:

$$MDL = t_{99\%CI} * s$$

where:
$t_{99\%CI}$ = the Student's $t$ value at the 99% confidence interval
s = the sample standard deviation as calculated above

6.3.6.4 The MDL, standard deviation and Student's $t$ statistic for the appropriate number of replicates at the 99% CI are automatically calculated when using the Laucks MDL database for ≤7 data points.

6.3.7 Methodology Exceptions/Specifics

6.3.7.1 Wet Chemistry

6.3.7.1.1 The MDL for all titrimetric determinations is set as the value determined by 0.2 ml of titrant at the method specified titrant strength and sample aliquot size. This would include all tests such as versenate hardness, alkalinity, argentometric or mercurimetric chloride, titrimetric COD, etc. Karl-Fisher moistures would be an exception to this; the MDL is taken to the value determined by 0.05 ml of titrant, the method specified titrant strength, and sample size.

6.3.7.1.2 The MDL for all gravimetric residue determinations (total solids, total suspended solids, etc.) is set as the value determined by a weighing of 0.2 mg at the method specified sample size.

6.3.7.2 GC and Gas Chromatography/Mass Spectrophotometry

6.3.7.2.1 MDLs for GC/MS results will be processed per the normal 40 CFR protocol to calculate a statistical MDL value. Part of the requirement for performing

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MDLs is to analyze a standard at the concentration of the MDL to verify that the compound can be detected. Part of the detection requirements for GC/MS is the spectral confirmation of the compound, not just the detection of the primary quantitation ion. Because of the spectral confirmation requirement, many of the statistically derived MDLs have a higher probability of not being detected. This fact in combination with the large number of compounds makes preparation of individual parameter concentrations close to the calculated MDL difficult and costly. Pre-mixes of standards are prepared at the same concentration level for all compounds in the mix.

6.3.7.2.2 Therefore, as a requirement of this SOP, Laucks has adopted the practice of using the standard concentration used to verify the calculated MDL as the reported MDL. Attempting to report lower than the verified value would be unjustified.

6.3.7.2.3 For the SIM methods this does not apply.

6.3.7.2.4 Likewise, for PCB and other multi-peak GC analyses, pattern recognition may also dictate what can actually be determined. For either situation, analyst interpretation may be in order to confirm actual compound identification. Such interpretation must be noted in the data.

6.4 Reporting Limits

6.4.1 Reporting limits are set by the laboratory at a level, usually at least 3 times the MDL and most often even higher. The idea is that the set limit not only be detectable but acceptably quantifiable. Given the statistical nature of the MDL calculation and the fact that real-world samples often introduce unknown factors, interferences, etc. that may even further affect variability, much less accurate quantification, a reliable RL should be significantly higher than the MDL. Reported values below the RL are considered estimated and in general, this should be indicated either by flagging the reported value as estimated or at least providing narrative comment to that effect.

6.4.2 Reporting Limits are generally determined in one of four ways:

- Administrative decision
- Set equivalent to the Practical Quantitation Limit (PQL)
- Project Specific Requirements
- The low calibration standard

6.4.3 The administrative decision method is generally based on what the laboratory considers to be a limit which can be reported on a consistent and reliable basis. Values obtained from

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statistical determinations of MDLs, for instance, cannot always be confirmed by spectral identification, pattern matching, standard response, or analytical spike recovery. In this instance, the laboratory may choose an RL which is more readily identifiable as a level for which a compound can be so identified and reliably quantified. Administrative decision may also be considered to be a part of the PQL option.

6.4.4 The PQL option is set as a factor times the MDL. This factor may either be set forth in the published method or it may be set by the laboratory. The multiplier is rarely defined in the method but Laucks will commonly use at least 3 to 5 times the MDL or even higher in some cases. In order to be able to provide consistent and routine reporting limits, the laboratory will generally not reset PQLs when MDLs are re-determined unless the MDL changes by a factor of more than twofold.

6.4.4.1 If it is determined from the study that the reporting limits must be changed (i.e. the MDL is near to or exceeds the RL), the QA Officer and the supervisor, often in concert with the Laboratory Director and/or Technical Director, must meet to determine the appropriate course of action which will often require client contact. Reporting limits are intended to be at a level for which reliable identification and reasonably accurate quantitation can be obtained. This generally cannot be done when the RL is close to the MDL.

6.4.5 Project Specific RLs are derived from project requirements and are contractually agreed upon between the laboratory and the client. In any event, the agreed upon limits **cannot** be less than the MDL or IDL if the 99% confidence level that the analyte is actually present is to be maintained. As long as they are achievable (i.e. the requested RL is not lower than the laboratory MDL), Laucks will generally comply with the client's request for that particular project, although frequently flagging will be used to indicate values are estimated below the RL.

6.4.6 A low-level standard may sometimes be used to show that a specific RL which is below the laboratory-established level is achievable but care must be taken in these instances. Quantitation will still be unreliable and if the statistics show it does not fall within the 99% confidence interval for MDLs, even if the standard is detectable, values in that range may still be falsely positive or negative and estimated. In some cases, pattern or spectrum matching may help to confirm the presence of an analyte at very low levels but this still would not improve the reliability of the actual quantitation.

6.4.7 Frequently, the low calibration standard defines the RL. The decision to use this technique may be any combination of method specific requirements, laboratory decision, or project-specific requirements. In no case will the RL determined from the low standard be lower than the statistically determined MDL.

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6.4.8 Reporting Limits are configured in the LIMS system, even those set by project-specific agreement, in which case they are detailed in documents pertaining to that project. The actual laboratory reporting limits should not be changed without the express permission of QA, the Technical Director or Laboratory Director. Project-specific limits are communicated by the Project Manager. Note here that the EPA Contract Laboratory Program (CLP) requirements use specific contract required detection limits (CRDLs) or quantitation limits (CRQLs) and any project using the CLP methods will almost always also be reported using the CLP CRDLs or CRQLs.

6.5 Precision and Accuracy (P&A) Studies

6.5.1 At a minimum, a one-time demonstration of precision and accuracy (P&A) must be performed for each method.

6.5.2 In most cases, it is also be required that an analyst perform a P&A study to be considered proficient and capable of independently performing a preparation or analysis.

6.5.3 P&A studies will be performed in accordance with the specific method. Where method-specific performance criteria are not specified, Laucks may choose to set criteria independently. Laucks' criteria, at a minimum, will meet those specified in a given method. Any determination to the contrary must be well documented and in direct consultation with QA and laboratory management.

6.5.4 All P&A studies must be turned in to QA after having undergone supervisory or senior analyst review. QA will review and sign the studies and in most cases return them to the supervisor for inclusion with their analyst and/or method files. If used to qualify an analyst, these studies will usually be accompanied by an Initial Demonstration of Capability form as described by NELAP and outlined more specifically in Laucks SOP LTL-1004.

6.5.5 All P&A studies must include the following information. This will be accomplished by using the P&A Information Sheet (See Appendix 3) and an Excel spreadsheet with the calculated P&A values and limits. The LIMS may eventually allow for all of the relevant information to be contained within a LIMS report in which case additional forms will not be necessary. As long as all of the relevant information is documented and can be tracked, the conditions of this SOP will be considered to have been met.

- Laboratory ID number
- Analyst who performed the preparation
- Method number of the preparation
- Date(s) the study was prepared
- Analyst who performed the analysis portion of the P&A study

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- Method number of the analysis
- Date(s) the study was analyzed
- Instrument name and ID which will distinctly identify that instrument; this cannot be a data "channel" from the computer system but must distinctly and uniquely identify that instrument.
- Spike level
- Measured concentration of the 4 replicates
- Standard Deviation of the recovery tabulated against the published QA Acceptance Criteria Table, where available
- Average recovery tabulated against the published QA Acceptance Criteria Table
- Concentration Units
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed
- Raw Data

6.5.6 The mean recovery and acceptance limits must meet the criteria given in the QC Acceptance Criteria Table at the end of each of the determinative methods, when available. Where criteria are not available Laucks may use internal acceptance limits or defer to a similar technical method with P&A criteria and use these as guidance in establishing performance criteria. In the case of organic SW846 methods, if the limits are not published in the individual method, the criteria in method 8000 (70%-130%) are followed as a guidance. In many instances, 70-130% is not achievable on a routine basis even by skilled staff. In this case, the laboratory (senior staff in conjunction with QA) may determine its own acceptance limits.

6.5.7 Blank spike analyses are the commonly accepted P&A evaluation. A mid-level spike processed through the complete analytical scheme is used to evaluate performance. In most methods where criteria are defined, 4 replicates must meet method-specified criteria for the **laboratory** to be considered capable of adequate performance. In general, reproducibility (%RSD) and recovery of the replicates are considered when evaluating performance.

6.5.8 The individual **analyst** must be able to analyze four replicates and meet laboratory blank spike control limits to be considered capable of performing the applicable analysis independently. For purposes of the P&A study, the analyst may be considered qualified if 90% of the analytes in a multi-analyte analysis meet laboratory criteria as long as all analytes meet the default method-specific criteria. As with the laboratory evaluation criteria, reproducibility (%RSD) of the replicates as well as recovery are considered when evaluating performance.

6.5.9 For the laboratory to be able to claim routine performance within specified limits, all analysts performing an analysis must be capable of that level of performance. All

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analysts must be routinely capable of performance within method-specified criteria and will be evaluated against laboratory criteria, with further action and training in order if they are unable to routinely meet laboratory limits.

6.6 Control Limits

6.6.1 Initially, when a new method is being implemented or there are insufficient data, the laboratory will use method-specified control limits for evaluation of data. If no such limits exist, the laboratory may elect to use specified limits from a similar method or may set default limits at the laboratory's discretion. These limits may be from the precision and accuracy study for that method. The determination for the suitability of setting any default limits not otherwise specified in a reference method is at the discretion of QA.

6.6.2 During the routine course of analysis, blank spike or laboratory control samples (LCS) and in many cases matrix spikes and matrix spike duplicates (or sample duplicates) will be analyzed. Spiking will occur at the levels specified in the respective methods where available, but will generally be somewhere in the middle of the calibration range.

6.6.3 When sufficient data have been gathered, generally at least 20 data points, the laboratory will undertake the determination of statistically-based control limits. These control limits are based on 3x the standard deviation of recoveries (for accuracy limits) or relative percent differences (for precision limits). In some instances, warning limits may also be established using 2x the appropriate standard deviation.

6.6.4 At a minimum, the control limits will be evaluated annually on a preparation/analysis/matrix specific basis. The number of data points and spiking levels used to obtain the new limits must be documented when forwarded to QA for approval.

6.6.5 If purchased from a commercial vendor, vendor-supplied control limits for a control sample will be considered adequate for default control limits if they are within the limits specified in the reference method. In addition, if the material is readily available and its composition does not change with every purchase, which is rarely the case, the laboratory will develop internal limits for that material. These limits may or may not be within the vendor-supplied limits but they **must** be within the method-specified limits.

6.6.6 Control limits may be specified in a reference method, in federal agency guidance documents or may be statistically determined by the laboratory from existing data. In general, laboratory determined limits for control samples must not exceed method specified limits. If laboratory determined limits do exceed method-specified limits, the entire system must be evaluated to improve method performance. In most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory cannot demonstrate adequate performance for all samples on a routine basis.

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6.6.7 Control limits may be specified in a reference method, may be statistically determined by the laboratory from existing data, or may be administratively set in project QAP or federal agency guidance documents. It is desirable that laboratory determined limits for control samples not exceed specified limits. However, for multi-component determinations that include analytes that are known to be poor performers, it may not be feasible to use non-statistically derived control limits. This will be observed most frequently for organic methods. As a consequence, the default 70-130% limits listed in SW 846 Method 8000, for instance, are frequently unreasonably narrow, as evidenced by many documents including the Department of Defense (DOD) Quality Systems Manual (QSM) and the Army Corps Shell document, all of which have statistical bases and are substantially wider than those in Method 8000. If laboratory determined limits do exceed method-specified limits, the system must be evaluated. Such an evaluation will include an assessment of the actual compound and comparison to control limit data from other sources (such as the DOD QSM). This evaluation should be documented on the spreadsheet or other paperwork associated with the control limit determination and any exceptions or special considerations discussed. It should be noted here that, for some compounds, no laboratory can consistently achieve the default method criteria.

6.6.8 That said still, in most instances, it is unacceptable for routine performance to exceed method-specified performance even if the laboratory is using method-specified control limits. This is because the laboratory's statistics would indicate that it cannot demonstrate consistent performance within the specified guidelines for all samples on a routine basis and that a QC sample that was within the specified limits would not necessarily indicate that the other related samples would also be within specifications. However, if the laboratory is clear about the internal limits that are being used, they are statistically based (rather than default values) and the user finds them acceptable, the laboratory may simply identify when the project or method limits are being exceeded and if they meet the internally calculated limits, the system will be considered as meeting the data quality objectives.

6.6.8.1 With particular concern regarding DoD limits as applied to organic methods for soil by extraction method 3545, a recent concern has arisen. The statistical derivation of the DoD limits was from data processed from control samples that were spiked after the addition of sodium sulfate, drying and transfer to the extraction cell. The most recent version of that method now requires spiking prior to addition of sodium sulfate. This has resulted in erratic recoveries which frequently fail to meet DoD recovery objectives. Laucks is currently working to resolve this issue either by obtaining a variance, contractual agreement or other course of action.

6.6.9 The laboratory may also calculate limits for matrix spike and matrix spike duplicate or replicate samples. However, these limits are primarily used to demonstrate method performance on a particular sample or sample-type relative to the routine laboratory

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sample and exceptions to these limits will generally be allowed as long as control sample limits are met.

6.6.10 The laboratory may be called upon to utilize control limits specified in a method or in a specific contract as designated in the LIMS database or in supplementary paperwork. The laboratory's overall performance will be considered adequate if internal control limits are within those specified in the reference method. Contractually defined limits will be used for the control samples analyzed under the contract and appropriate corrective actions taken but will not be used as a guide for routine laboratory performance.

6.6.11 For any particular project, if the laboratory exhibits exceptions to the method or contract-specified criteria, appropriate corrective action must be taken. Should routine laboratory control limits be within method or contract-specified criteria, and laboratory limits are exceeded but method or contract limits are met, the data may be reported but should be flagged. Where appropriate, corrective action may still be taken at the discretion of QA.

6.7 Laboratory Uncertainty

6.7.1 NELAP and DoD require that a laboratory estimate its measurement uncertainty for a particular analyte in a particular matrix but do not specify how that is to be accomplished. One approach is to determine the standard deviation of a laboratory control sample (blank spike in a clean matrix) and calculate the 95% confidence level as twice this value. This is equivalent to the blank spike warning limit from a control limit determination and is what Laucks uses to fulfill this requirement.

6.7.2 Note that this is **only** a measurement of **laboratory** uncertainty and does not take into account factors associated with sampling, matrix, transport, storage or other factors that may introduce additional uncertainties.

6.7.3 In order to avoid confusion, Laucks only reports this value if requested by the client.

## 7. Reports

7.1 Data Package Organization

7.1.1 All work, with the exception of control limit computations, is performed under laboratory SDG ID numbers.

7.1.2 All data supporting the study are provided in a standard format specific to that method. In order to save paper, some items, such as the initial calibration, etc., may be referenced to other SDGs. However, it must all be easily recoverable if full documentation is required, up until the standard laboratory data disposal date. Rationalizations for

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interpreting the results of any study and specific detail which might impact the study should be documented in the file as well.

7.1.2.1 Data files are prefaced with a copy of the summary report containing all of the elements previously noted in this SOP. Where laboratory database reports are available, a copy of the database report must also kept on file by QA. All sign-offs will be handwritten.

7.1.2.2 All documents pertaining to a study are to be scanned (paper) or saved electronically (spreadsheets, etc.) in the appropriate iLIMS SDG and/or run sequence files locations.

## 8. References

40 CFR Part 136, Appendix B, *Definition and Procedure for the Determination of the Method Detection Limit, Revision 1.11*

EPA Contract Laboratory Program (CLP) Inorganics Statement of Work (SOW), ILM04.2.

EPA Contract Laboratory Program (CLP) Inorganics Statement of Work (SOW), ILM05.3.

EPA Contract Laboratory Program (CLP) Organics Statement of Work (SOW), OLM04.3

EPA Contract Laboratory Program (CLP) Organics Statement of Work (SOW), SOM01.1

EPA "600" Series Methods, section 8.1.1, 40 CFR, Part 136, App. A.

EPA SW846 "8000" Series Methods, both the general method 8000B and the specific methods

*National Environmental Laboratory Accreditation Program (NELAP)*, 2003 Standard

*Department of Defense Quality Systems Manual*, Final Version 3, January 2006

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## Appendix I

### Student's *t* Values

| n | degrees of freedom | *t* value at 99% CI |
|---|---|---|
| 2 | 1 | 31.821 |
| 3 | 2 | 6.965 |
| 4 | 3 | 4.541 |
| 5 | 4 | 3.747 |
| 6 | 5 | 3.365 |
| **7** | **6** | **3.143** |
| 8 | 7 | 2.998 |
| 9 | 8 | 2.896 |
| 10 | 9 | 2.821 |
| 11 | 10 | 2.764 |
| 12 | 11 | 2.718 |
| 13 | 12 | 2.681 |
| 14 | 13 | 2.650 |
| 15 | 14 | 2.624 |
| 16 | 15 | 2.602 |
| 17 | 16 | 2.583 |
| 18 | 17 | 2.567 |
| 19 | 18 | 2.552 |
| 20 | 19 | 2.539 |
| 21 | 20 | 2.528 |
| 22 | 21 | 2.518 |
| 23 | 22 | 2.508 |
| 24 | 23 | 2.500 |
| 25 | 24 | 2.492 |

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**Appendix II**

**MDL Spreadsheet**

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Instrument: HPLC5 (Oscar)
Column: Varian C18
Matrix: Water
Prep method: SW3535 Solid Phase Extraction (SPE) (3535(8330))
Analysis method: Nitroglycerin by High Performance Liquid Chromatography (8332)
DeterminationID: 11523
Run sequence ID: 14440
Units: ug/L

MDL Study: 14440-1

| AnalyteID | Analyte | MDL 1 | MDL 2 | MDL 3 | MDL 4 | MDL 5 | MDL 6 | MDL 7 | MDL 8 | MDL 9 | MDL 10 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 894 | Nitroglycerin | 1.0562 | 1.09138 | 0.95515 | 1.08071 | 0.96779 | 1.06047 | 1.10238 | | | |
| 976 | PETN | 0.49838 | 0.5067 | 0.4461 | 0.49089 | 0.4843 | 0.52343 | 0.53221 | | | |
| 1659 | EGDN | 0.8906 | 0.95853 | 0.98733 | 0.92652 | 0.87704 | 0.96923 | 0.88863 | | | |

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MDL Study: 14440-1

Instrument: HPLC5 (Oscar)
Column: Varian C18
Matrix: Water
Prep method: SW3535 Solid Phase Extraction (SPE) (3535(8330))
Analysis method: Nitroglycerin by High Performance Liquid Chromatography (8332)
Determination ID: 11523
Run sequence ID: 14440
Units: ug/L

| Analyte ID | Analyte | #Reps | True Value | Mean | % Rec | Std Dev | t-Value | MDL | Mean/MDL | %RSD | CV | PQL | C1 | C2 | C3 | C4 | C5 | C6 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 894 | Nitroglycerin | 7 | 1 | 1.044869143 | 104.4869143 | 0.05932562 | 3.143 | 0.186460424 | 5.6 | 5.7 | 17.5 | 2.5 | P | + | P | P | P | P |
| 976 | PETN | 7 | 0.5 | 0.497430429 | 99.48608571 | 0.02834919 | 3.143 | 0.089101504 | 5.58 | 5.7 | 17.5 | 1.2 | P | + | P | P | P | P |
| 1659 | EGDN | 7 | 1 | 0.928267 | 92.8267 | 0.04415953 | 3.143 | 0.138793403 | 6.69 | 4.8 | 20.8 | 2.5 | P | + | P | P | P | P |

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MDL Study: 14440-1

Instrument HPLC5 (Oscar)
Column: Varian C18
Matrix: Water
Prep meth SW3535 Solid Phase Extraction (SPE) (3535(8330))
Analysis m Nitroglycerin by High Performance Liquid Chromatography (8332)
Determinat 11523
Run seque 14440
Units: ug/L

t-values

| samples | Student-t value |
|---|---|
| 4 | 4.521 |
| 5 | 3.747 |
| 6 | 3.365 |
| 7 | 3.143 |
| 8 | 2.998 |
| 9 | 2.896 |
| 10 | 2.821 |

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MDL Study: 14440-1

Instrument HPLC5 (Oscar)
Column: Varian C18
Matrix: Water
Prep meth SW3535 Solid Phase Extraction (SPE) (3535(8330))
Analysis m Nitroglycerin by High Performance Liquid Chromatography (8332)
Determinat 11523
Run seque 14440
Units: ug/L

* = Measured value <35% of true value
#Reps = Number of replicates used in the calculation
Mean = (sum replicates)/#Reps - computed via iLIMS 'lilengine' calculation engine
% Rec = (Mean * 100)/(True Value)
Std dev = SQRT((sum replicates)^2) - (mean^2)/#Reps))/(#Reps - 1) - computed via iLIMS 'lilengine' calculation engine
t-Value = Student's t-value used for calculation, based on #Reps; see t-Values worksheet for tabulated values
MDL = t-Value * (Std dev)
%RSD = (100 * (Std dev))/mean
CV = mean/(Std dev)
PQL = PQL from determination definition
C1 = P-->MDL <= (True Value); *-->test failed
C2 = P-->(Mean/MDL) <= 5;+-->5 < (Mean/MDL) <= 10; *-->test failed
C3 = P-->MDL*3 < PQL; *-->test failed
C4 = P-->#Reps >= 6; *-->test failed
C5 = P-->35 <= % Rec <= 150; *-->test failed
C6 = P-->True value * 10 <= PQL; *-->test failed

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**Appendix 3**

**P&A Information Sheets**

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# Laucks Testing Laboratories, Inc.
# Precision and Accuracy Study Information Sheet

This form must be submitted in addition to a spreadsheet summary of the spiking level, measured concentrations including units of measurement, average and standard deviation of the recovery tabulated against published QA acceptance criteria, and the supporting raw data.

SDG Number: _____

**Analysis:**

Analyst: _____

Analysis Method: _____

Analysis Date: _____

Instrument ID: _____

**Preparation:**

Prepared by: _____

Preparation Method: _____

Preparation Date: _____

Cleanup Method(s): _____

**Review:**

Analyst's Signature: _____ Date: _____

Supervisor's Signature: _____ Date: _____

QA Approval: _____ Date: _____

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1012

**Title: Solvent QC Monitoring for Trace Residue Analysis**

Revision history:

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|---|---|
| 1 | 4/10/91 |
| 2 | 4/27/99 |
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| 4 | 9/20/07 |

Revised by: Harry Romberg, Quality Assurance Officer Date: 9/20/07

Reviewed by: Barbara O'Neill, Extractions Manager Date: 9.20.07

Approved by: Hugh Prentice, Laboratory Director Date: 9-20-07

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## 1. Introduction and Scope

1.1 Description

1.1.1 The purpose of this SOP is to define the method(s) used to check and document the purity of the major solvents used for trace residue analysis at Laucks. The solvents being tested are methylene chloride, acetone, and hexane. Specific techniques and equipment used for operations such as concentration and solvent exchange are not addressed in this document.

## 2. Equipment List and Reagents

2.1 Equipment and Reagents

2.1.1 Other Glassware, reagents and equipment as delineated in the methods specific to the described task.

## 3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Typical precautions should be taken when handling any solvent. Some, such as methylene chloride are not flammable, but others, such as acetone and hexane are and should be treated with extreme caution. Long term health effects of solvent contact are often unknown but generally unfavorable. Breathing of ANY solvent vapor should be minimized, as should any direct skin contact, by working in a well ventilated area (in or near a hood if necessary) and by using the provided gloves and, if necessary or desired, respirator masks.

3.2 Waste Disposal

3.2.1 All waste solvents should be disposed in the appropriate waste solvent container, never in the sink or in combination with aqueous liquids.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on hazardous waste disposal.

## 4. Operation procedures

4.1 Sequestering of Solvent

4.1.1 A new lot of acetone or methylene chloride must be sequestered by the supplier and the checking process initiated at least four weeks prior to using up the last of the previous lot of that solvent. A lot is defined as a batch of solvent with the same manufacturers lot

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number. This must be done in order to ensure that the lot has been released for analytical use BEFORE the remainder of an acceptable lot has been used up. If any solvent has failed, a second bottle may be tested for the failed parameter(s) in order to ensure the failure was not due to laboratory contamination. Failure of the second test is grounds to reject that lot for use in the laboratory.

4.1.2 When a lot has been formally designated as acceptable, enough should be ordered to last approximately 2 months in order to minimize the frequency of testing necessary. Typically, the supplier will ship 10 cases of a sequestered lot of methylene chloride at a time and acetone as requested. No more than 4 months supply of methylene chloride will ever be ordered, as typical methylene chloride recommended shelf-life is 6 months.

4.1.3 Alternatively, since it is unlikely that any lot will fail and to eliminate the time between acceptance and delivery, an appropriate supply (as defined above) may be ordered and sequestered at the laboratory for analysis. This is commonly done for hexane since we do not consume large quantities of this solvent. If said lot fails, however, the lot must be returned to the supplier and a new lot tested immediately. This lot MUST be kept separate from the current stock and very clearly marked so that it is not inadvertently used prior to acceptance. This distinction is the responsibility of the Extractions Supervisor. All solvent deliveries must be immediately reported to the Extractions Supervisor or designated alternate in order that this distinction be made.

4.2 Initiation, Data Handling and Record Maintenance

4.2.1 The Extractions supervisor or designated representative initiates the checking process. For any month in which any extractions solvent or other reagent QC is performed, a LIMS SDG is created under the "*Organic Extractions QC*" project. When a bottle from a new, previously untested lot of solvent is received, a sample ID number is assigned from the next available ID in the SDG. This sample ID must contain the manufacturer, lot number, solvent, and tests (test codes) to be performed. See Appendix I for examples.

4.2.2 The three solvents which we specifically check in the extractions lab are methylene chloride, acetone, and hexane. The test codes used for these purposes are *"ACS Methylene Chloride Acidity"*, *"8270 Solvent Checks"*, and *"8081 Solvent Checks"*. These codes are used **only** for testing solvents and for nothing else. Other checks, such as sodium sulfate or Florisil should use the test code appropriate for the analysis for which the material will be used (8081, 8270, etc.)

4.2.2.1 Methylene chloride is checked for acidity, semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are all 3 specifically noted above.

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4.2.2.2 Acetone is checked for semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are *"8270 Solvent Checks"* and 8081 *"8081 Solvent Checks"*.

4.2.2.3 Hexane is checked only for pesticides. The respective test code assigned to this analysis is *"8081 Solvent Checks"*.

4.2.3 When testing has been completed, the lot will be officially designated as acceptable or failed by the QA Officer or Technical Director. This will be done by initialing the final report and sending a copy to the Extractions supervisor. Any lot will be considered acceptable which meets the criteria specified in Appendix II. The Extractions supervisor should be certain that a lot has been designated as acceptable prior to using it and should take whatever actions are necessary to ensure prompt analysis and acceptance before the last of the acceptable solvent has been used.

4.2.4 The data and report files will be maintained by the respective extractions and analysis departments as they would anyt other SDG. Extractions will maintain the solvent check files so that they can always be assured that the solvents they are using have been checked.

4.3 Solvent Analysis

4.3.1 Methylene Chloride Acidity

4.3.1.1 0.01 N NaOH - To a 100 mL. volumetric flask, add 10 mls. of .1000 N sodium hydroxide from the buret of standardized NaOH in the Inorganics lab. Fill to the volumetric mark with deionized water, stopper, and mix very well. It takes several inversions of the flask to properly mix the solution (at least 10). This solution should be prepared immediately prior to analysis.

4.3.1.2 Neutral ethanol - Add 25 mls. of denatured ethanol to an Erlenmeyer flask. Add 2 or more drops of phenolphthalein indicator solution (1 gm. phenolphthalein/100 mls. ethanol). With a Pasteur pipet, add the 0.01 N NaOH solution dropwise until the ethanol turns slightly pink. Hold the flask against a white background to enhance the color. This solution should be prepared immediately prior to analysis

4.3.1.3 Add 25 mls. of the methylene chloride to be checked to the flask containing neutralized ethanol. Swirl. Do not shake too vigorously so that $CO_2$ from the air will not acidify the ethanol and cause a fading endpoint.

4.3.1.4 Add 900 µL of the 0.01 N NaOH. Swirl to mix well.

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4.3.1.5 If the resulting color is pink, the methylene chloride passes (is not acidic). If it does not turn pink, it should be retested, preferably from a second bottle. If it fails a second time, it should be rejected or used only for cleaning. Failing solvent should NEVER be used for extraction purposes.

4.3.1.6 A "PASS" or "FAIL" is entered into the iLIMS report under the associated regular test code, *"ACS Methylene Chloride Acidity"*. If the solvent fails, residue analysis SHOULD NOT be performed until a suitable acceptable lot is determined. The Extractions supervisor should see that any such failing lot has been terminated in the LIMS. The report, however, should still be submitted to the QA Officer.

4.3.2 Residue Checks

4.3.2.1 The residue checks are performed for EPA CLP Target Compound List (TCL) components for both pesticides/PCBs and semivolatiles (ABNs) as is appropriate for the solvent being checked.

4.3.2.2 In all cases, varying amounts of the appropriate solvent is concentrated to 1 mL. in a Kuderna-Danish (KD) concentrator. No splitting of the concentrate occurs. Surrogates are not added.

4.3.3 Methylene Chloride - $MeCl_2$ is used for both ABN and pesticide/PCB analyses. A separate 250 mL. concentration is done for each analysis.

4.3.3.1 For the pesticide/PCB analysis, hexane is added and the solvent exchanged and concentrated down to 1.0 mL., which is submitted for analysis.

4.3.3.2 For ABN analysis nothing is added and the $MeCl_2$ concentrated directly down to 1 mL and submitted for analysis.

4.3.4 Acetone - Acetone is used for both ABN and pesticide/PCB analyses. A separate 40 mL. of 1:1 acetone/$MeCl_2$ concentration is done for each analysis.

4.3.4.1 For the pesticide/PCB analysis, hexane is added and the solvent exchanged and concentrated down to 1.0 mL., which is submitted for analysis.

4.3.4.2 For the ABN analysis, the acetone is blown down to near dryness with nitrogen and brought up to 1 mL. with $MeCl_2$ and submitted for analysis.

4.3.5 Hexane - Hexane is used only in pesticide analysis. It will be concentrated 25 mls. to 1 mL. as stated and submitted for TCL pesticide/PCB analysis.

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4.4 Reporting and Acceptance

4.4.1 Acceptance criteria are compiled in Appendix II and are based on the appropriate amounts of solvent concentrated to 1 mL. final volume. They are derived from Laucks reporting limit criteria for acceptable blanks. The LIMS report indicates the acceptance level, the level found and signifies whether the detected level (if any) passes (OK) or fails (FAIL).

4.4.2 The analyst should also look at the chromatogram and determine if there are other peaks or humps that might interfere with detection or quantitation of target analytes. Should the solvent display any interferences, either target or non-target analytes, it should be rejected. The analyst should indicate in the test report whether or not the solvent passes this subjective judgment. The individual checking the data should confirm this interpretation and reporting.

4.4.3 If any of the checks fails, the QA Officer and Extractions Supervisor must be notified immediately. It may be that other analyses can be terminated and the solvent rejected or that a second analysis for the failing fraction begun without wasting the resources to complete the other tests.

4.4.4 After the analyst has reported the data in the LIMS and the data has been checked, the QA Officer or designated individual will review the entire report and sign-off on the acceptance or rejection of the solvent. A copy of the initialed approval or rejection and test report is given to Extractions for filing and to readily document the status of each solvent lot.

4.4.5 Approved and signed-off solvent may be released to the lab for immediate use. Arrangements should be made immediately to return any unopened failing solvent and to use opened containers for cleaning or rinsing or in another manner that does not contribute to sample analyses.

4.5 Data Package Organization

4.5.1 A copy of the signed acceptance form along with the raw data is retained under the assigned laboratory SDG. The Extractions supervisor keeps a photocopy of the signed approval sheet in a file in the extractions lab.

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# Appendix I

## ILIMS Solvent Check Screens

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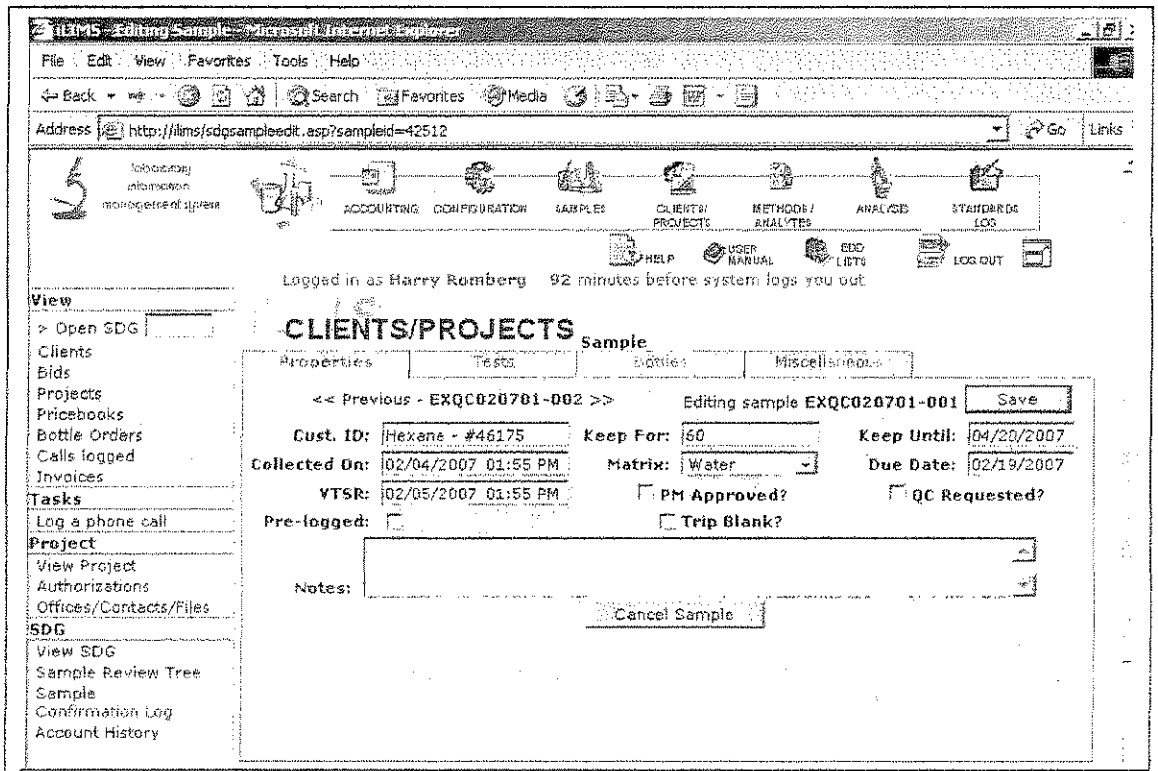


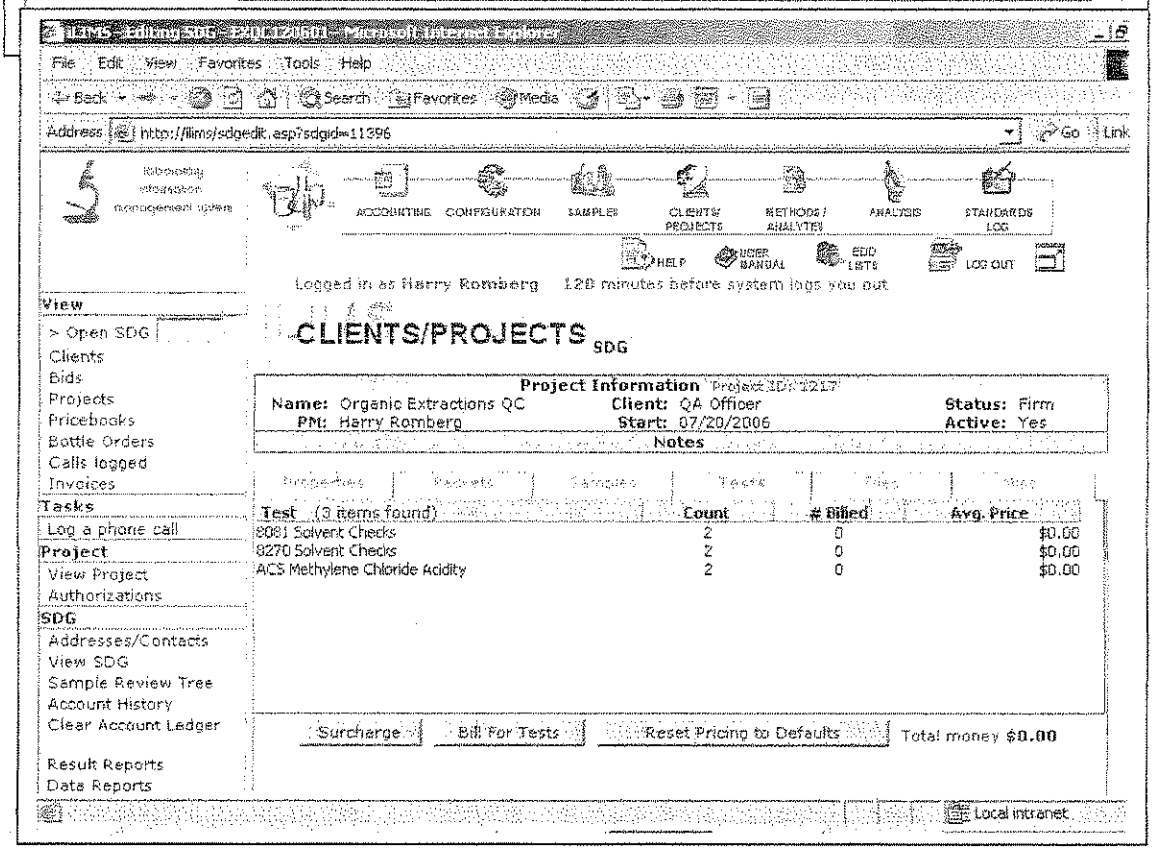

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# Appendix II

## Solvent Acceptance Criteria

| Semivolatile Compounds | Total ng in 1 mL. |
|---|---|
| Phenol | 2500 |
| bis(2-Chloroethyl) ether | 2500 |
| 2-Chlorophenol | 2500 |
| 1,3-Dichlorobenzene | 2500 |
| 1,4-Dichlorobenzene | 2500 |
| 1,2-Dichlorobenzene | 2500 |
| 2-Methylphenol | 2500 |
| 2,2'oxybis(1-Chloropropane) | 2500 |
| 4-Methylphenol | 2500 |
| N-Nitroso-di-n-propylamine | 2500 |
| Hexachloroethane | 2500 |
| Nitrobenzene | 2500 |
| Isophorone | 2500 |
| 2-Nitrophenol | 2500 |
| 2,4-Dimethylphenol | 2500 |
| bis(2-Chloroethoxy)methane | 2500 |
| 2,4-Dichlorophenol | 2500 |
| 1,2,4-Trichlorobenzene | 2500 |
| Naphthalene | 2500 |
| 4-Chloroaniline | 2500 |
| Hexachlorobutadiene | 2500 |
| 4-Chloro-3-methylphenol | 2500 |
| 2-Methylnaphthalene | 2500 |
| Hexachlorocyclopentadiene | 2500 |
| 2,4,6-Trichlorophenol | 2500 |
| 2,4,5-Trichlorophenol | 2500 |
| 2-Chloronaphthalene | 2500 |
| 2-Nitroaniline | 2500 |

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| Semivolatile Compounds | Total ng in 1 mL. |
|---|---|
| Dimethylphthalate | 2500 |
| Acenaphthylene | 2500 |
| 2,6-Dinitrotoluene | 2500 |
| 3-Nitroaniline | 2500 |
| Acenaphthene | 2500 |
| 2,4-Dinitrophenol | 5000 |
| 4-Nitrophenol | 2500 |
| Dibenzofuran | 2500 |
| 2,4-Dinitrotoluene | 2500 |
| Diethylphthalate | 2500 |
| 4-Chlorophenyl-phenyl ether | 2500 |
| Fluorene | 2500 |
| 4-Nitroaniline | 2500 |
| 4,6-Dinitro-2-methylphenol | 2500 |
| N-nitrosodiphenylamine | 2500 |
| 4-Bromophenyl-phenylether | 2500 |
| Hexachlorobenzene | 2500 |
| Pentachlorophenol | 2500 |
| Phenanthrene | 2500 |
| Anthracene | 2500 |
| Carbazole | 2500 |
| Di-n-butylphthalate | 2500 |
| Fluoranthene | 2500 |
| Pyrene | 2500 |
| Butylbenzylphthalate | 2500 |
| 3,3'-Dichlorobenzidine | 2500 |
| Benzo(a)anthracene | 2500 |
| Chrysene | 2500 |
| bis(2-Ethylhexyl)phthalate | 2500 |
| Di-n-Octylphthalate | 2500 |
| Benzo(b)fluoranthene | 2500 |
| Benzo(k)fluoranthene | 2500 |
| Benzo(a)pyrene | 2500 |
| Indendo(1,2,3-cd)pyrene | 2500 |
| Dibenz(a,h)anthracene | 2500 |
| Benzo(g,h,i)perylene | 2500 |

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## Solvent Acceptance Criteria

| Pesticide/PCB Compounds | Total ng in 1 mL. |
|---|---|
| alpha-BHC | 25 |
| beta-BHC | 25 |
| delta-BHC | 25 |
| gamma-BHC | 25 |
| Heptachlor | 25 |
| Aldrin | 25 |
| Heptachlor epoxide | 25 |
| Endosulfan I | 25 |
| Dieldrin | 50 |
| 4,4'-DDE | 50 |
| Endrin | 50 |
| Endosulfan II | 50 |
| 4,4'-DDD | 50 |
| Endosulfan sulfate | 50 |
| 4,4'-DDT | 50 |
| Methoxychlor | 250 |
| Endrin ketone | 50 |
| Endrin aldehyde | 50 |
| alpha-Chlordane | 25 |
| gamma-Chlordane | 25 |
| Toxaphene | 2500 |
| Aroclor-1016 | 500 |
| Aroclor-1221 | 1000 |
| Aroclor-1232 | 500 |
| Aroclor-1242 | 500 |
| Aroclor-1248 | 500 |
| Aroclor-1254 | 500 |
| Aroclor-1260 | 500 |

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-1013

**Title: Preparation, Storage, Shelf Life and Traceability Documentation of Standards, Reference Materials and Stock Reagent Chemicals**

Revision history:

| Number | Date |
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| 2 | 4/17/96 |
| 3 | 6/3/96 |
| 4 | 9/12/00 |
| 5 | 8/11/04 |
| 6 | 12/7/05 |
| 7 | 5/8/07 |

Revised by: Harry Romberg Date: 5/8/07
Harry Romberg, QA Officer

Approved by: Hugh Prentice Date: 5/8/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP is intended to describe the way in which standards and reference materials are tracked, prepared, stored and maintained at Laucks, from the time of receipt of the neat or stock materials, solutions or their preparation to the point of use of the working standard. Though general descriptions of documentation of standard preparation may be present, it is not intended to define the actual method of preparation for each specific method. This is contained in the applicable analytical method SOP. The way in which these standards are tracked, however, is detailed along with the description of storage and shelf life guidance. Reagent chemical shelf-life, documentation and labeling requirements are also discussed, though documentation and tracking requirements are not quite as rigorous as for standard materials.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described procedure of documentation.

1.2 Definition of Terms

1.2.1 Standard or Reference Material: these items are defined as any solution of an analyte at a known concentration prepared from purchased neat materials or stock solutions, or from intermediate solutions traceable to purchased materials. This includes calibration standards, independent laboratory control standards (LCS or SRM), spiking solutions, surrogate solutions, independent calibration verification standards, etc. Although these may be prepared or used in different ways and may differ in how they are used in the iLIMS database, the tracking systems, though through different modules, are much the same.

1.2.2 Reagent Chemical: Any chemical used by the laboratory whether used as a standard or in the detection or quantification of another constituent. A reagent, sometimes in conjunction with other reagents, reacts with the target analyte and can thus be used to determine the presence and even the concentration of the target species. In the context of this SOP, reagents can have shelf-lives beyond which they may degrade and no longer be useful for their intended purpose.

1.2.3 Shelf Life: The period of time a solution my be held under proper storage conditions before it is no longer considered valid. Materials which have exceeded their respective shelf lives must not be used for quantification purposes.

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1.2.4 Expiration Date: The date after which a standard solution is no longer considered valid for quantification purposes. Such a standard is defined as having exceeded its shelf life. Although the expiration date does not either guarantee that a standard is still good if the current date is prior to expiration or that it is bad immediately after expiration, these dates are administrative and must not be exceeded even by 1 day or subsequent data for all but qualitative analyses will most often be considered invalid on this basis alone.

## 2. Equipment Lists, Reagents and Standards

2.1 Equipment

2.1.1 Equipment and reagents necessary for the preparation of any specific solution.

- All volumetric pipets and flasks must be Class A.
- Mechanical pipets (i.e. Eppendorf, etc.), calibrated at the prescribed frequency and within tolerance (generally ±2%)
- Syringe pipets – those currently in use have a stated tolerance of ±1%, though Laucks considers ±2% as adequate for most purposes

2.2 Reagents

2.2.1 Reagents may either be prepared by the lab from raw chemicals or may be purchased solutions. The preparation and occasionally even the shelf-life may be specified by the manufacturer. Preparation and use from the point of purchase are detailed in the specific analytical SOP.

2.3 Standards

2.3.1 Standards as specified in each analytical SOP.

2.3.2 All standards must also be verified both qualitatively and quantitatively in order to satisfy EPA requirements for traceability. This may be accomplished by either (1) purchasing solutions which have been fully documented by a commercial vendor, or (2) following the recommended steps for traceability as outlined in the CLP Organic statement of work, OLM04.2, Exhibit E, Section 5. *Note: The latter method is not typically used but is allowed if necessary.*

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2.4 Computer with network and iLIMS database access

2.5 Standard/Reagent Chemical Logbooks – These logbooks are usually electronic

## 3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards and reference materials including neats or solutions should be handled as if they are hazardous substances.

3.2 Waste Disposal

3.2.1 Waste segregation and disposal from the point of collection is further covered in Laucks SOP on Hazardous Waste Disposal Procedures

## 4. Operation

4.1 Reagent Chemical Practices

4.1.1 Laucks recognizes that some reagent chemicals deteriorate over time and may not be suitable for certain purposes, particularly for use as standards or where deterioration will affect the efficacy of a particular analysis. On the other hand, many chemicals do not change over a very extended period of time and may be useful for years or even decades.

4.1.2 Therefore the following practices have been adopted for chemicals which are **not** being used as standards. Standard materials are addressed elsewhere and those practices take precedence over those used for bulk reagent chemicals.

4.1.2.1 All chemicals must be dated **BOTH** upon receipt and opening.
Received: mm/dd/yyyy
Opened: mm/dd/yyyy

4.1.2.2 If the chemical is one already present in the laboratory and has not been marked with these dates, the label may be marked as "dates of receipt and opening unknown, before [today's date]".

4.1.2.3 All chemicals with expiration dates provided by the manufacturer should be assumed to expire on that date and disposed appropriately.

4.1.2.4 Chemicals with no listed expiration date may be labeled "does not expire". However, care should still be taken when using such materials. In fact, certain

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conditions should be met even if the chemical DOES have an expiration date which has not yet passed. If they are observed to have discolored they may have deteriorated beyond usefulness. Excessive clumping or absorption of water may have led to their deterioration and if this will affect the purpose for which the reagent will be used, a newer reagent should be selected. Some chemicals may be reconstituted or used by first heating or drying but only where this will not have a deleterious or unknown effect upon its usefulness.

4.1.2.5 In some cases, a material may be assayed to determine if its purity remains adequate for its intended use. As long as it continues to be sufficiently pure for any purposes for which the chemical may be used, it may be retained.

4.1.2.6 If a chemical does NOT meet the purity or age requirements for one purpose but DOES remain adequate for other purposes, it may be labeled "do not use for the analysis of xxxx" or "to be used only for yyyy" or in some other way marked that it may only be used in limited circumstances.

4.2 Preparation of Organics and Inorganics Materials

4.2.1 General consideration in standard preparations include:

4.2.1.1 Determine volumes and aliquots required using the concentration calculations in Appendix 1.

4.2.1.2 Choose volumes and aliquots which minimize the number of intermediate dilutions required to obtain final working concentration considering:

- The inherent measurement error, i.e. no aliquots less than 20% of the volume of measurement device whenever possible.
- The ratio of solvent:analyte
- The amount of solution left over for disposal.

4.2.1.3 Be sure to use a solvent volume sufficient to dissolve all analytes.

4.2.1.4 The solvent used should be miscible with water when being used for sample spiking purposes. Most standards used in the organic extractions laboratory are prepared with methanol or acetone.

4.2.2 Proper Syringe/Pipette Technique

4.2.2.1 Choose an appropriate size syringe so that the measured volume is at least 2/3 of the total volume of the measurement device.

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4.2.2.2 When selecting a pipette, choose volumetric pipettes only for the exact amount to be measured.

4.2.2.3 Always rinse a syringe (organics) at least 3 to 5 times with the appropriate solvent in between measurements, and wipe the syringe with a Kim-wipe.

4.2.2.4 There should be no air bubbles. Either tap them away or discard the solution in the syringe/pipette and obtain another aliquot. Repeat this procedure as often as necessary to remove all bubbles. It may be helpful to use a GC septum with very small (<50 µl) syringes.

4.2.2.5 For organics, when delivering the measured volume to the dilution vessel, fill the vessel 1/2 - 2/3 with the solvent to be used, add the measured aliquot directly into the solvent without touching the sides of the container, and fill to volume with solvent. A sub-surface injection is preferable whenever possible.

4.2.3 Proper Micropipet Technique

4.2.3.1 Mechanical or syringe pipets should be calibrated at the frequency and as described in Laucks SOPs LTL-8006 entitled *"Organics Department Micropipet Calibration"* or LTL-9003 entitled *"Micropipet Calibration."*

4.2.3.2 Generally micropipets are only used for dispensing aqueous solutions. Non-aqueous solutions may not dispense accurately. Syringe pipets may be used to dispense either aqueous or organic solutions.

4.2.3.3 The appropriate sized micropipet is selected and a new appropriate tip attached.

4.2.3.4 The piston is depressed to the first stop, the tip inserted into the liquid to just beneath the surface and the piston slowly released so that the tip fills but does not aspirate up into the barrel of the pipet. Care must be taken to keep the tip below the surface for the entire aspiration.

4.2.3.5 After the material is aspirated, the tip is removed from the liquid. It should be lightly touched to the side of the container to remove any liquid sticking to the outside of the pipet tip. Since volatile materials are not dispensed with these pipets, subsurface dispensation is not necessary.

4.2.3.6 The tip is then introduced into the receiving vessel and the piston depressed to the to the first and then to the second stop. It is often best to touch the tip to the side of the receiving vessel to make sure there is no drop hanging onto the tip. When the piston is released, the analyst should also be aware if there is any residual liquid sticking to the inside of the pipet tip. This would indicate incomplete dispensation.

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The piston may need to be depressed again in order to accurately deliver the entire contents. The resulting solution should then be thoroughly mixed in the receiving vessel.

4.2.4 When preparing stock solutions from neats, the following steps should be taken.

NOTE: 99.9% of the time, stock standards will be prepared WEIGHT per Volume. DO NOT use Volume measurements for liquids unless EXPRESSLY TOLD to do so by your SUPERVISOR.

4.2.4.1 The dilution vessel (volumetric flask) and stopper should be triple solvent rinsed (last time with the solvent to be used for standard preparation) and allowed to dry completely.

4.2.4.2 The neat is weighed, to 4 significant figures, directly into the volumetric flask and the weight is recorded (to 3 decimal places for volatiles, one less than actually weighed in order to account for possible small losses due to volatilization). Stopper before weighing to avoid compound volatilization if dealing with solvents or volatile materials.

4.2.4.3 For components other than volatiles, the volumetric flask is filled about 3/4 full with dilution solvent and shaken until analyte is completely in solution.

- If the analyte will not dissolve, the stoppered volumetric flask should be sonicated in the sonic bath or thoroughly mixed with a vortex mixer until it does dissolve. (Because sonication heats the solution slightly, the solution should be allowed to cool before dilution to the mark). Consult your supervisor if the compound is not in solution after sonication.
- The volumetric flask is diluted to the mark.
- If the analyte recrystallizes while stored in the refrigerator, the standard should be sonicated before use. Do not aliquot from a cloudy or opaque standard.
- In addition to the normal labeling of the standard, a separate label should be added indicating the need for sonication.

4.2.4.4 For volatiles, the flask is inverted and gently mixed three times after diluting to the mark. The solution in the neck of the flask is then discarded before use.

4.3 Traceability Documentation for Organic and Inorganic Standards

4.3.1 All standard materials are logged into the iLIMS database when they arrive in the lab. All analysts have access to this level of the database. No neat material should be used before it has been logged into the database. Examples of some iLIMS standards database screens are provided.

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4.3.2 Standards which are intended to be used as independent secondary calibration checks, lab control samples, or standard reference materials must be logged into the SRM database. Any materials that will be used in this manner can only be logged in by select individual supervisors or QA. Any other analyst must inform one of these individuals so that the material can be properly logged in and therefore become available for use in constructing iLIMS batches. Some materials may need to be logged into both the standards and SRM databases. See Section 4.4 for additional detail.

4.3.3 The current controlled logbooks are identified in each area as follows. More books may be added from time-to-time if necessary for whatever reasons. As they are all kept together in one database system, there should be no chance of confusion, damage or loss of a particular book.

| Name | Department |
|---|---|
| AP | Conventional Chemistries |
| EX-10 | Organic Extractions |
| HA1 | GC Semivolatiles |
| HERB1 | GC Semivolatiles |
| HPLC-1 | HPLC |
| IC-7 | Conventional Chemistries |
| IN-3 | Conventional Chemistries |
| IOM-3 | Conventional Chemistries |
| MA-8 | GC Semivolatiles |
| ME-15 | Metals Preparation |
| MS-9 | GC/MS ABNs |
| MV-8 | GC/MS Volatiles |
| Neats | Organics |
| PX-8 | Pesticides/PCBs |
| REA2 | Inorganics |
| Reagents-1 | Organic Extractions |
| SC-Neats1 | Special Chemistries |
| SC1 | Special Chemistries |
| TE-7 | Conventional Chemistries |
| TOC-4 | Conventional Chemistries |
| VOA-5 | GC Volatiles |

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**NOTE:** The numbers in the above table indicate a sequential number, beginning with 1, with each subsequent controlled book with that analysis code having the next higher integral value. This is a carry-over from the old system when there were physical logbooks. This system will accommodate any number of logbooks or departments. Unless these logbooks get large and complex, there should be no need to create new logbooks with the same letter designations. However, if for whatever reason the same letter designation is used, the next sequential number must be used as well.

4.3.4 All purchased neats, stocks and subsequent standard preparations must be recorded in the appropriate log-book.

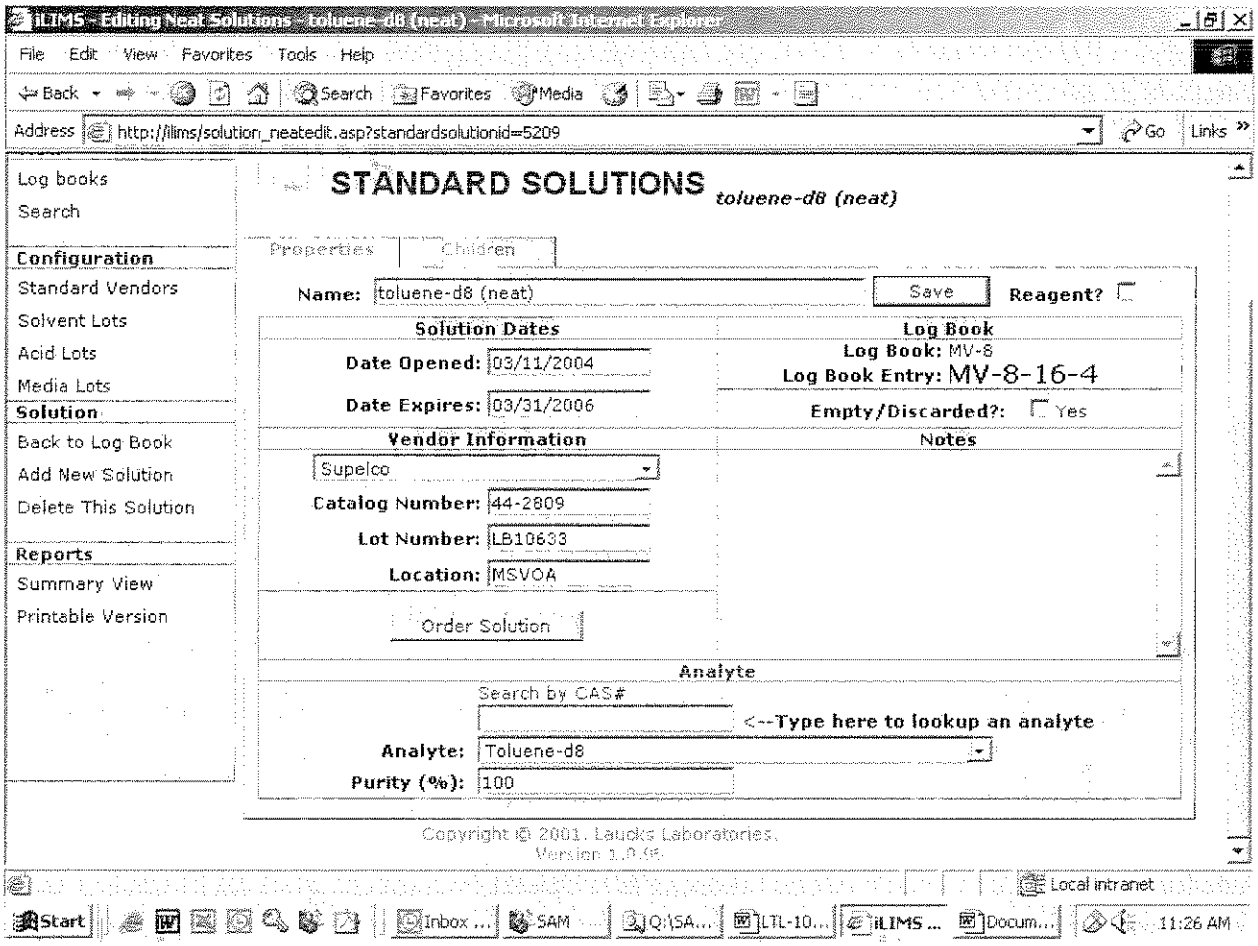
4.3.5 Materials must be consistently named so that each and every identifier/analysis for which that solution will be used are delineated (i.e. a gasoline standard must be identified as gasoline, GRO, Gasoline Range Organics, etc. as all of these terms may be used for different analysis methods, even though it is the same solution.)

4.3.6 Upon receipt, each purchased neat material, purchased stock solution or in-house prepared solutions are entered into a standards log-book and assigned a unique LAUCKS identification number. Each of these types of solutions have a slightly different configuration in the LIMS but at a minimum contain certain elements of information. Following are the "properties screens for each of these types of solutions.

**NOTE:** When naming any solutions either in this section or the following section regarding Standard Reference Materials (SRMs) and Laboratory Control Samples (LCSs), it is best to use a consistent mnemonic naming system as this will greatly facilitate finding them later when they are actually used in batches and when they need to be looked-up in the database again. For instance, if all ICP calibration standards have the word "trace" in their name, the operator can filter the list of available solutions from a logbook using the word "trace" and get a much shorter list from which to select.

Anyone entering stocks or SRMs should also avoid using unnecessarily long names which may run off of the pick list screen, electronic disk deliverable field, or other listing.

4.3.7 Neat Screen:



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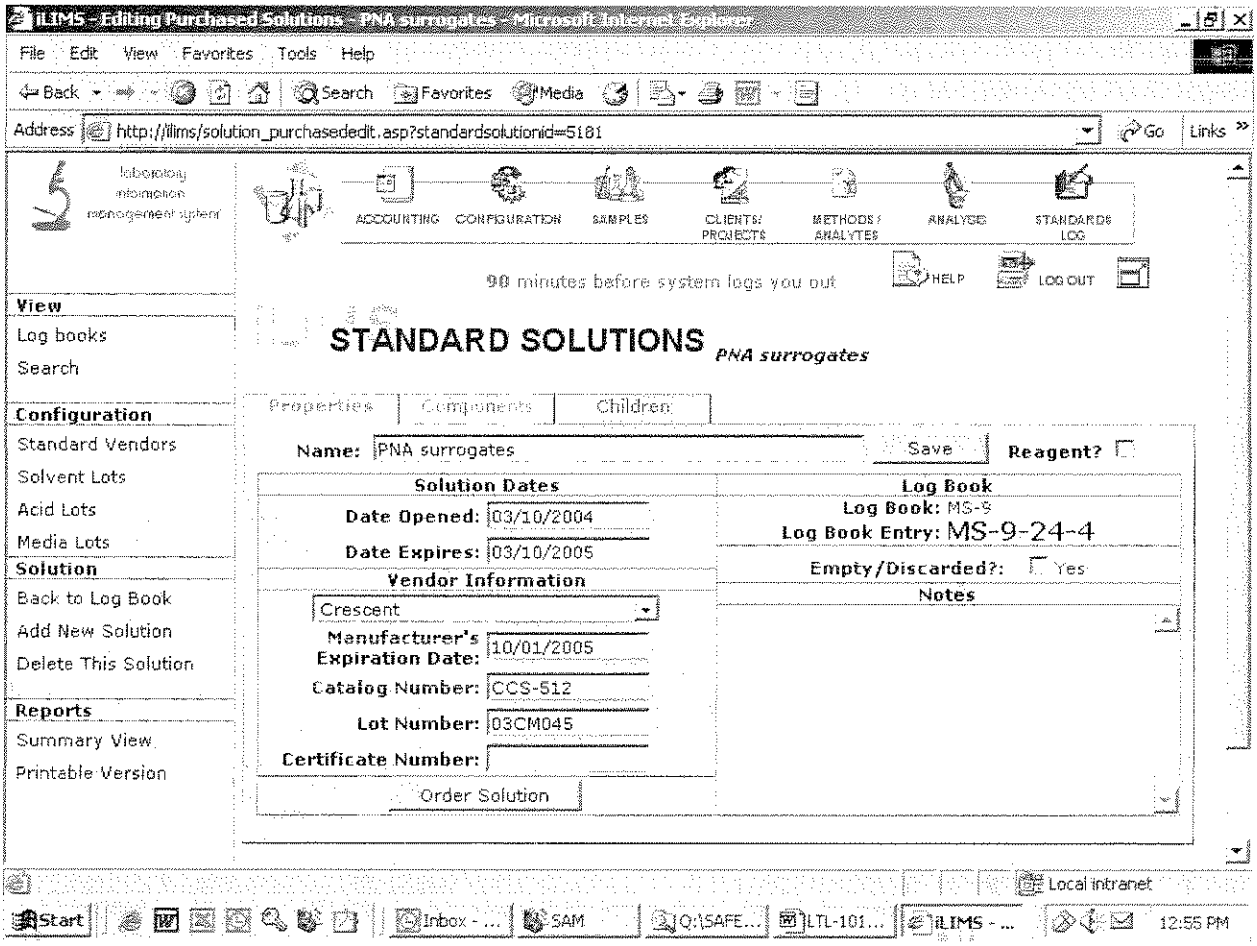
Purchased Solution Screen:



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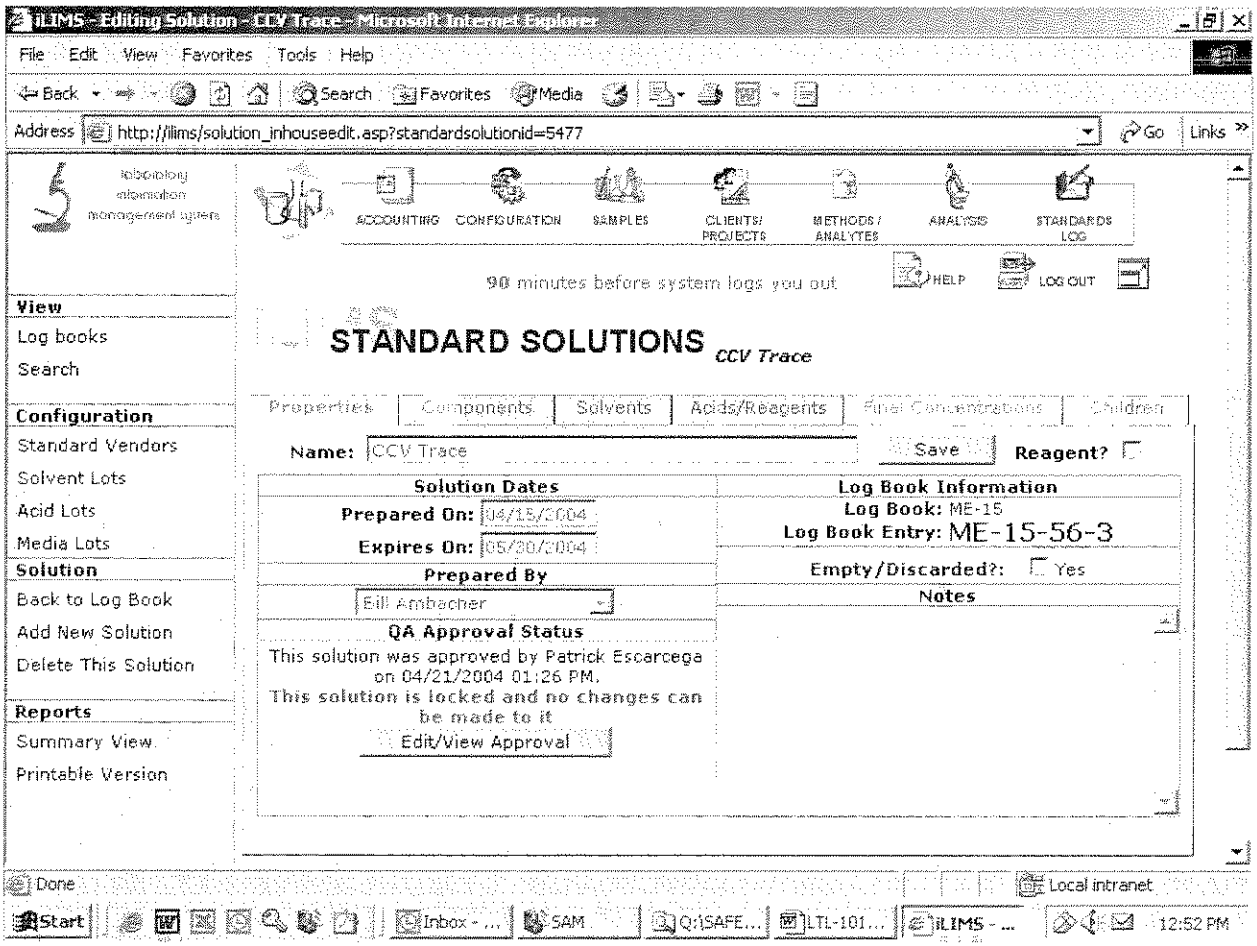
In-house Screen:




4.3.8 All elements of these property screens should be filled out as completely as possible particularly with preparation and expiration dates, vendor or preparer's names and, in the case of purchased materials, lot numbers.

4.3.9 The expiration date is either vendor supplied, in the analytical SOP or determined from the shelf life table in Appendix 2, in order of preference.

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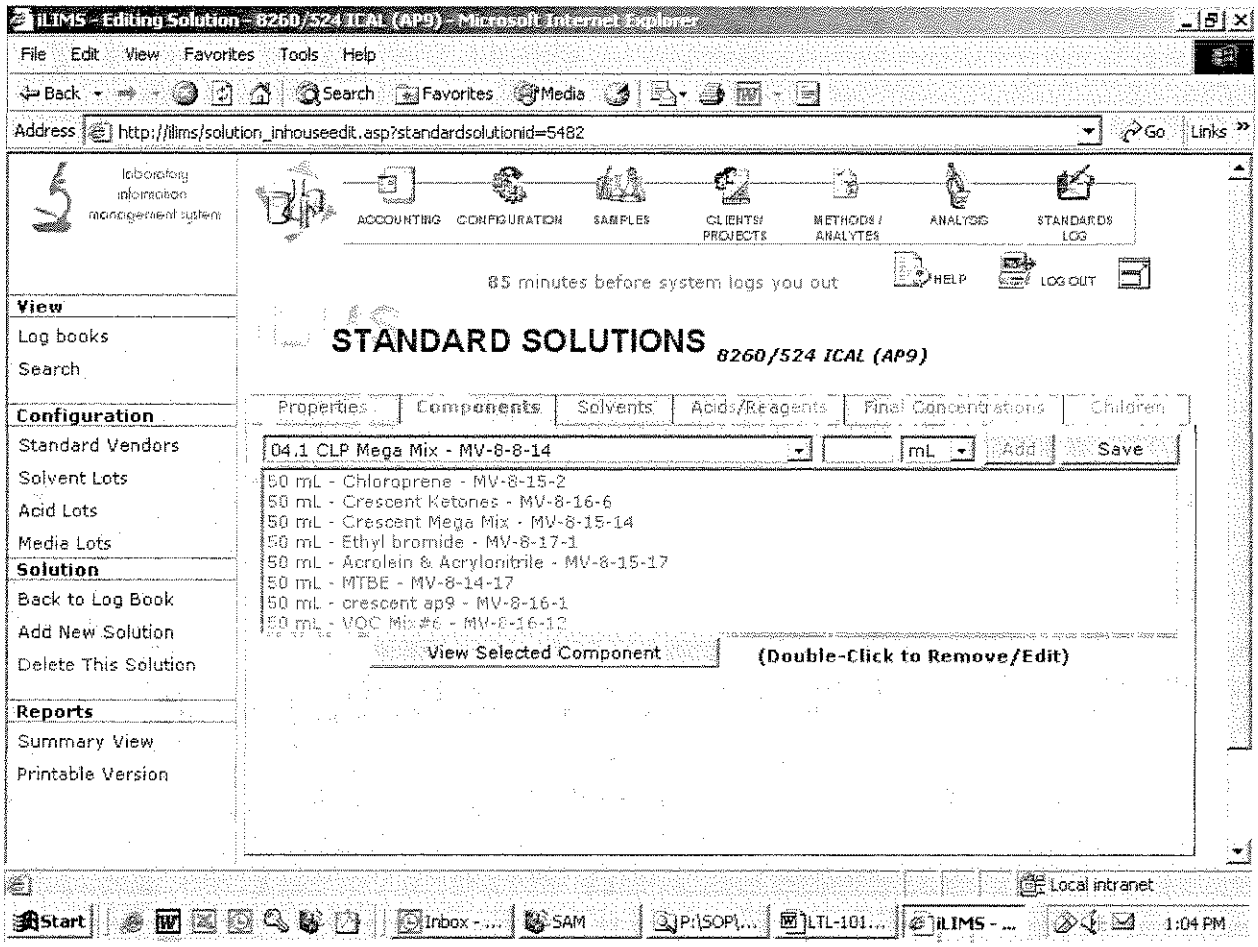
4.3.10 After one of these screens are completed, the component screen must be filled in.



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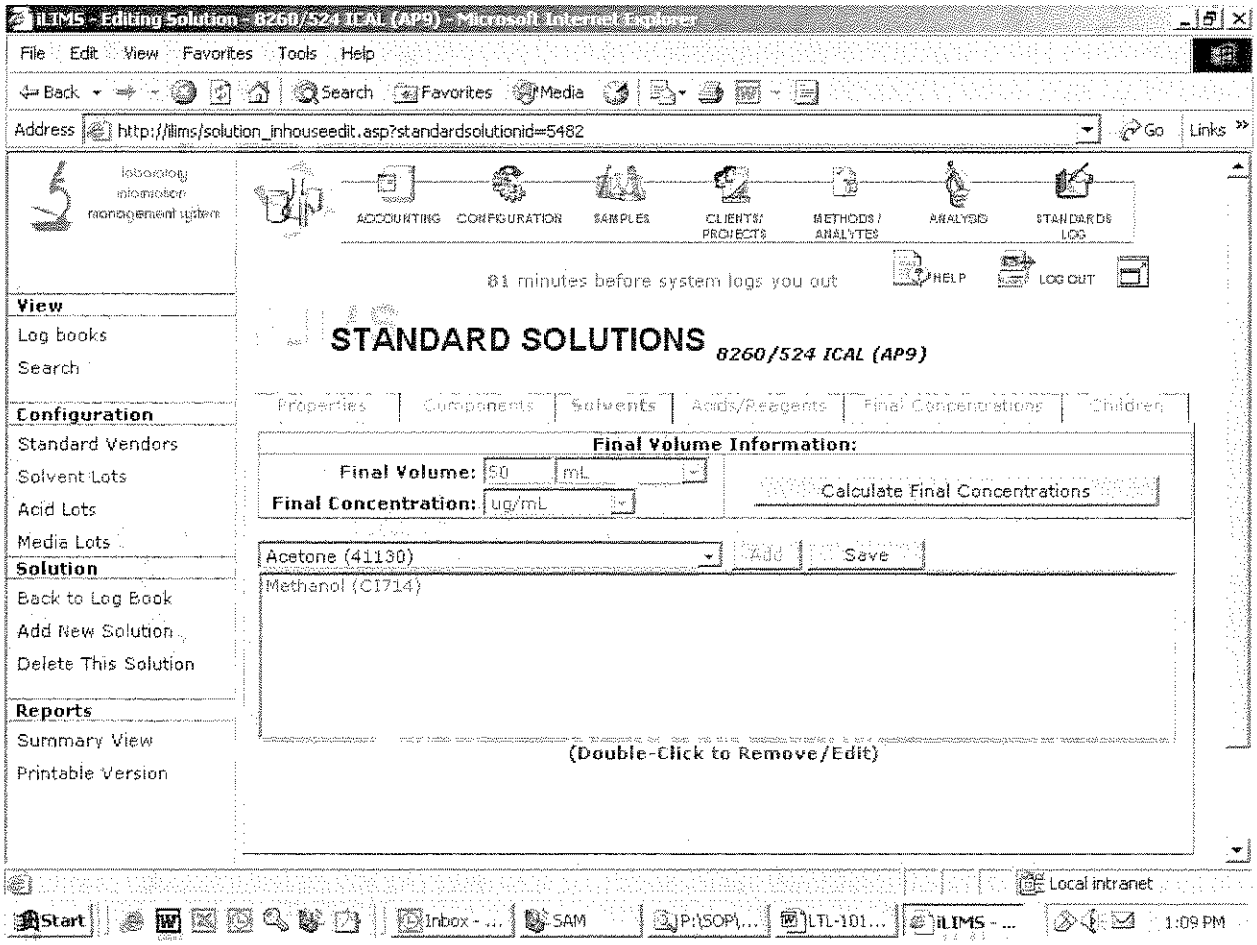
4.3.11 Materials which are prepared from neats or purchased solutions will need to have the "solvent" screen filled in. This will help the program to determine the final concentration of the components and to track solvent lots used to prepare the standards.



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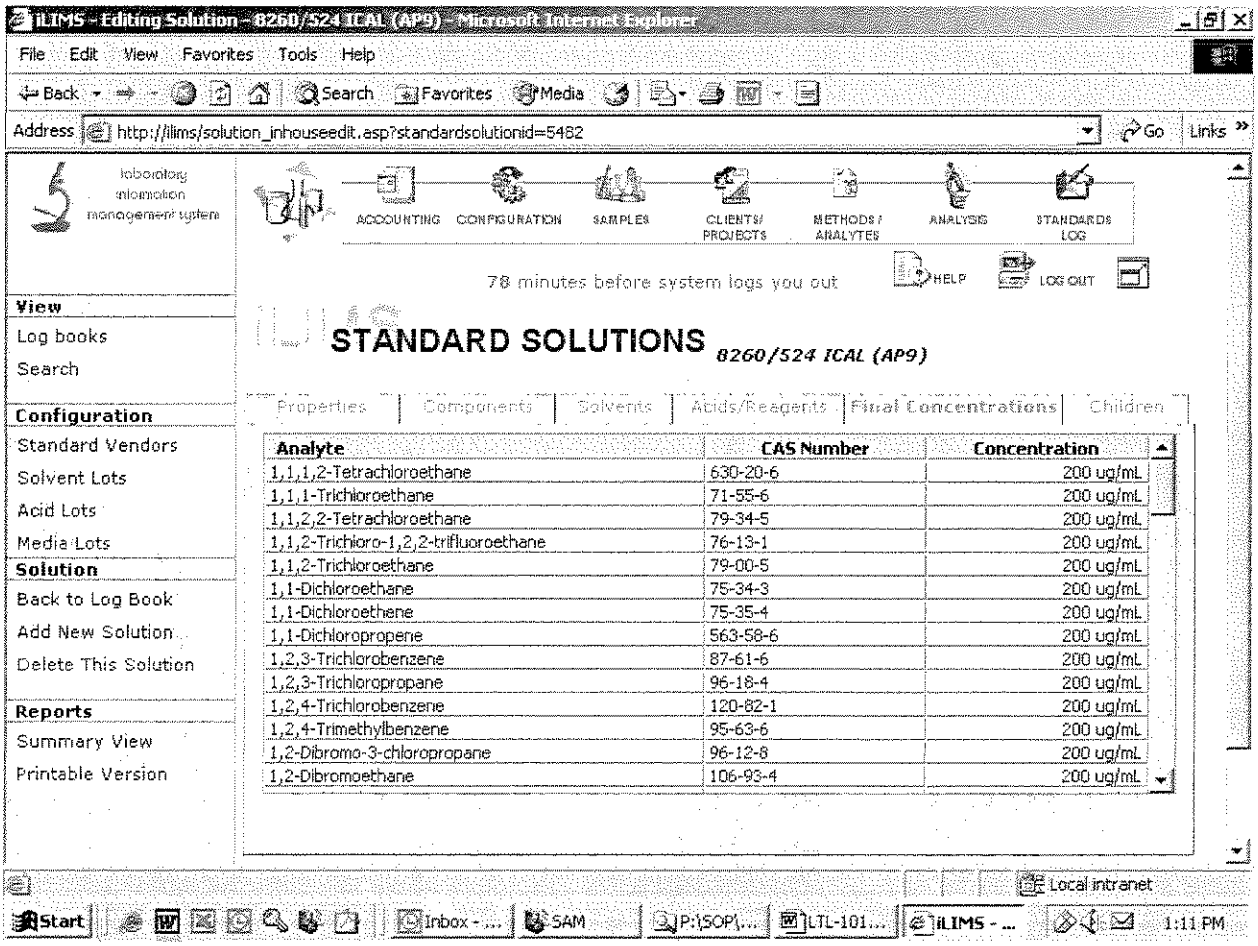
4.3.12 This will result in the final concentration screen to be completed.




| Analyte | CAS Number | Concentration |
|---|---|---|
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 200 ug/mL |
| 1,1,1-Trichloroethane | 71-55-6 | 200 ug/mL |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 200 ug/mL |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1 | 200 ug/mL |
| 1,1,2-Trichloroethane | 79-00-5 | 200 ug/mL |
| 1,1-Dichloroethane | 75-34-3 | 200 ug/mL |
| 1,1-Dichloroethene | 75-35-4 | 200 ug/mL |
| 1,1-Dichloropropene | 563-58-6 | 200 ug/mL |
| 1,2,3-Trichlorobenzene | 87-61-6 | 200 ug/mL |
| 1,2,3-Trichloropropane | 96-18-4 | 200 ug/mL |
| 1,2,4-Trichlorobenzene | 120-82-1 | 200 ug/mL |
| 1,2,4-Trimethylbenzene | 95-63-6 | 200 ug/mL |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | 200 ug/mL |
| 1,2-Dibromoethane | 106-93-4 | 200 ug/mL |

4.3.13 After each material is logged it is labeled with the LAUCKS ID, date received, date opened (if the material is to be used from the same container more than once) and expiration date (if not already on the label). The accompanying vendor Certificates of Analysis, Purity or Authenticity are labeled with the Laucks ID and filed in a controlled laboratory notebook in the laboratory area. These certificates are then archived through QA when the notebook is full.

4.3.14 The nomenclature used by the database to assign database ID numbers derives from the old physical logbook system but continues to be useful for assigning ID numbers which can be easily tracked back through the system. Using a working ABN standard prepared on 4/7/04 as an example. The solution number assigned was MS 9-25-2. This label represents the following:

- MS - solution was made and used as a semivolatile mass spec standard
- 9- solution was logged into standard book #9

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- 25- page number on which solution has been recorded
- 2- this denotes the second entry on page 25

4.3.15 All discrete measurements made during a standard preparation must be recorded in the logbook/database, specifically, weights aliquots and final volumes.

4.3.16 Other pertinent data to be in the log book are as follows but other pieces of information may also be recorded but are not currently required:

- Standard Name
- Parent material and concentration/purity
- Solvent, acid or other diluent standard is prepared in and lot number where appropriate
- Type of standard being prepared (i.e. neat, purchased stock or in-house prepared)
- Final concentration
- Date prepared/opened in all cases
- Expiration dates in all cases
- Analyst's name, if prepared and not simply purchased

4.3.17 The Laucks internal working material ID must be documented on the manual benchsheet, the analytical run-log or instrument printout to enable tracking back to the parent material.

4.3.18 Laucks iLIMS training modules are available for individuals who have not worked with the iLIMS standards logbook before.

4.3.19 In addition, there are several short "How-to" documents describing how to use specific aspects of standards documentation. These are currently available by navigating to the User Manual at the top of the standard iLIMS screen. Clicking the "bookmarks" tab opens the table of contents where can expand the Standard Log bookmark and find the topic of interest. Those available at the time of this writing are as follows and should be read by anyone to whom the specific situation applies. Copies of these current versions are located in Appendix 4.

- Converting Molarity or Normality to Concentration
- Co-eluting and Degradation Compounds
- Standards With Non-concentration Units
- Using the Purity Factor

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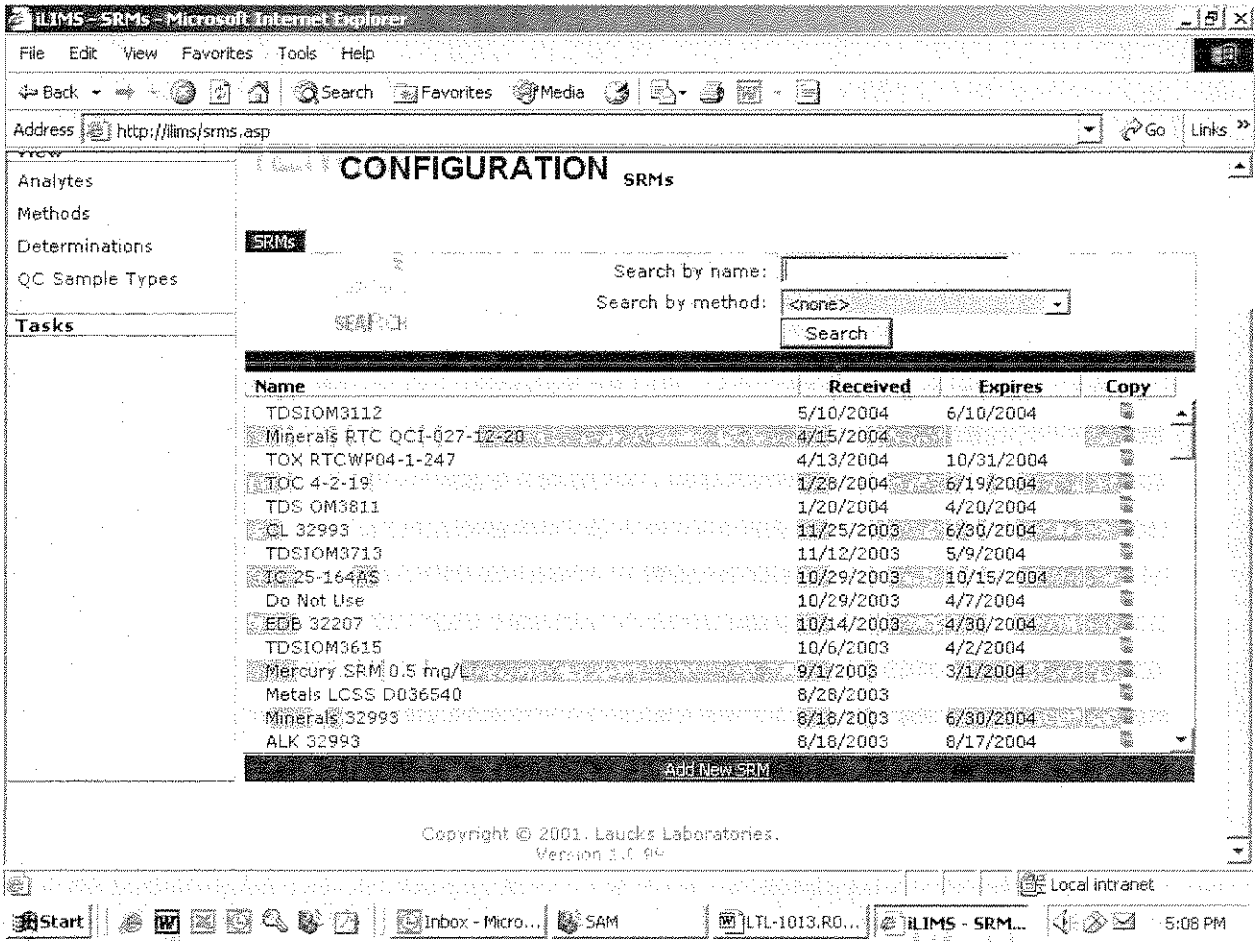
4.4 Traceability Documentation for Organic and Inorganic Laboratory Control Samples (LCS) and Standard Reference Materials (SRM)

4.4.1 Only select individual supervisors or QA have access to this part of the iLIMS database. Other individuals must request one of these people to create database entries in order to make these solutions available for QC batches.

4.4.2 SRM Master Database Screen




4.4.2.1 The easiest way to create a new SRM if one already exists is to use the copy function and then to modify the entry created. This also enables one to keep similar naming conventions with all relevant SRMs which in turn makes them easier for users to find. Otherwise, one must create an SRM using the "add new SRM" key and create a name which is relevant with enough detail to be easily discernable but succinct enough to be useful.

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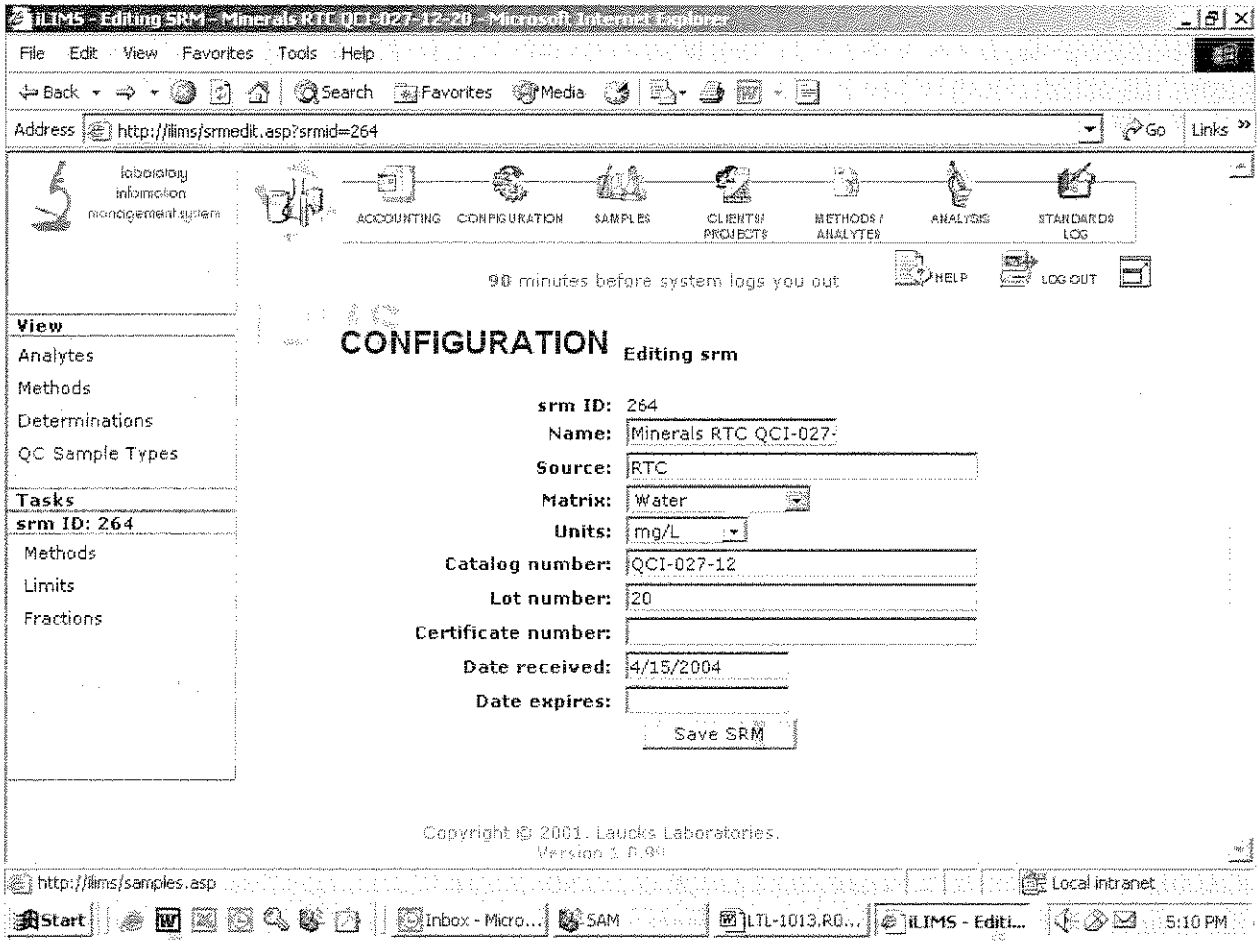
4.4.3 SRM Configuration Screen




4.4.3.1 One then edits or creates the appropriate SRM information. All known information about the SRM should be completed paying particular attention to the name, lot number and expiration date, saves the SRM and then must review the limits section (see 4.4.4).

4.4.3.1.1 The expiration date is set at the manufacturer's expiration date, if one is supplied. If the SRM is made from an old PT sample which does NOT have an expiration date, which is generally only done for a limited number of inorganic constituents, a period of one year **from receipt** is used, unless there is reason to expect it to be less. If the material is made from stock chemical, a similar time is used from the **preparation** date.

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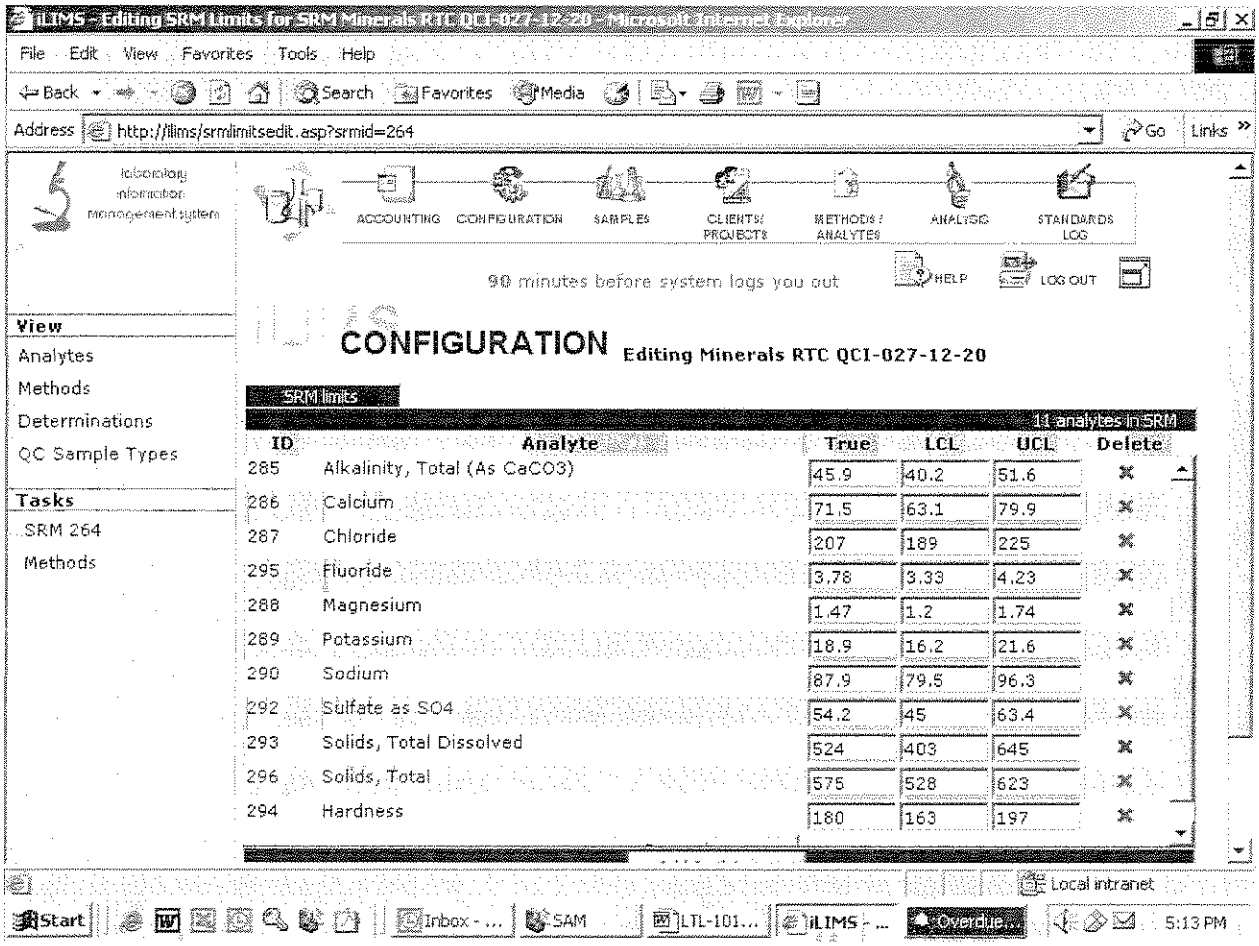
4.4.4 SRM Limits Screen




4.5 Storage of Standards and Reference Materials

4.5.1 Organic Standards and References Materials

4.5.1.1 Store in vial or bottle which minimizes head space.

4.5.1.2 Use amber or clear glass, screw tops with Teflon-liners when required, and store at, in order of preference, the temperature referenced in the analytical SOP or the temperature detailed below, in the assigned refrigerator or freezer.

4.5.1.3 Volatile Standards and Reference Materials

4.5.1.3.1 All standards solutions should be stored in the VOA freezer at -10°C to -20°C.

4.5.1.3.2 Most volatile standards are stored in flame-sealed ampules until used.

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4.5.1.3.3 Standards are transferred to Mininert vials with Teflon lined septa for daily use and stored in the VOA freezer. When the standards are transferred, the information is recorded in the GC/MS Volatile Standards log book in the iLIMS database.

4.5.1.4 Other Volatile Standard Solutions

4.5.1.4.1 Some standards need to be prepared in the lab. Stock solutions are diluted using high purity MeOH.

4.5.1.4.2 To insure stability, standard solutions should be sealed in amber glass ampules

4.5.1.4.3 Rinse unsealed ampules with clean MeOH and place in oven to dry.

4.5.1.4.4 Cover ends of ampules with foil.

4.5.1.4.5 Dilute stock solution in high purity MeOH in a volumetric flask.

4.5.1.4.6 Mix gently.

4.5.1.4.7 Partially fill ampules with solution and recap with foil.

4.5.1.4.8 Use $CO_2$ to cool ampules until crystals form on sides.

4.5.1.4.9 Heat end of ampule with acetylene flame until glass begins to soften.

4.5.1.4.10 Gently pull end until seal is formed.

4.5.1.4.11 Label ampules and store in freezer.

4.5.1.4.12 Record the information in the Mass Spec VOA Standards Log Book (MV) in the iLIMS database.

4.5.1.4.13 When standard solutions are used they should be transferred to Mininert cap vials with Teflon lined septa properly labeled as previously discussed. The vials are stored in the VOA freezer until discarded.

4.5.1.5 Semivolatile Standards and Reference Materials

4.5.1.5.1 All standards solutions should be stored at a maximum temperature of 4°C±2°C. Refer to the analytical SOPs for details as some analytes may drop out of solution if at cooler temperatures.

4.5.2 Inorganic Standards and Reference Materials

4.5.2.1 All metals standards are kept in a cabinet in the metals analysis lab. This is at room temperature. Expired standards that are kept for qualitative purposes are kept in the same room, in a different cabinet. These qualitative standards have a special label on the bottles denoting that they are not to be used for quantitative purposes. All other (non-metals) standards are kept at 4°C±2°C in a reach-in cooler in the

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inorganics lab. This cooler is dedicated to standards and SRMs only. No sample storage is allowed in this cooler.

4.6 Shelf Life

4.6.1 Expiration

4.6.1.1 If a parent material has an expiration date of month/year, then the material is considered usable through the end of that month. For example, 01/07, the material expires after 1/31/07. This guidance was obtained from various vendors.

4.6.1.2 All parent expiration dates MUST be entered into the standards database and the expiration date for all resulting child materials must also be entered into the database and placed on the material label.

4.6.1.3 Note that no child solution may exceed the life of a parent solution or neat material. This stipulation may reduce the shelf life of a prepared solution from that listed in Appendix 2. For instance, if a stock solution is prepared from parent material that has an expiration date of 5/20/07 in 1/07, instead of having a six month shelf life (07/07) the solution will expire, 05/20/07, the same date as the parent.

4.6.1.4 See Appendix 2 for the Table of typical shelf life of standards and reference materials. This table is provided as guidance only. The vendor expiration date (if applicable) and the analytical SOP take precedence over any guidance set forth in the Table.

4.6.1.5 Although not a preferred practice, from time to time an expired standard may be used for specific non-quantitative purposes. If a standard is past its expiration date it may be used for qualitative purposes only. An additional label must be placed on the standard. This label must be bright in color and must clearly indicate that it is to be "Used for Qualitative Purposes Only". Any sample results other than simply detected or undetected should be clearly labeled as qualitative only.

## 5. Standard Verification

5.1 Criteria

5.1.1 Most stock standards purchased from reputable vendors are certified to NIST standards. However, it is still prudent practice to verify that an incoming stock is the material and concentration it is certified to be. So, wherever possible, the laboratory should verify incoming stock against known materials already in the laboratory, even if those older materials have already expired, in order to prove the standards are what they are supposed

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to be. ANY actual standard verification should include both the standard approved and the standard against which it was compared.

5.1.1.1 Criteria for standards acceptability are in many cases defined in individual SOPs. In instances where they are not so defined, acceptability criteria compared to an independent source are:

- 80% - 120% for organics
- 90% - 110% for inorganics

5.1.2 Even though the stock and child standards are logged into the iLIMS standards database and include the concentrations and amounts used in child standard preparation, a documented physical comparison of standards is still highly desireble. It is also required that the standards be reviewed and approved by a supervisor or their designee, preferably not the individual who prepared and/or entered the standard into the database. This approval will lock the standard in the database so that it cannot be further edited except by approved individuals. The approval also makes the standard available for inclusion in iLIMS run logs, etc.

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## Appendix 1

## Example Calculations

### 1. Concentration Calculations from Neat Materials

Note: If purchasing a certified (NIST traceable) stock solution from a vendor, these steps and some of those in the second section can be ignored. One only needs to calculate dilutions of the original stock.

HELPFUL hint: To keep yourself straight ALWAYS, ALWAYS include the units (mg, ml, etc.) in your calculations.

Example Calculations of Standard Concentrations:

Weight of Neat Material: 0.2500 gm
Volume of Solvent: 10 ml

To Calculate Concentration in mg/L (ppm):

1) Calculation in Steps.

A) $$0.2500gm * \frac{1000mg}{1.0gm} = 250mg$$

A.1) Adjust the 250 mg for purity,

i.e. if purity = 90%, 250 mg x 0.9 = 225 mg

Although the iLIMS will handle any purity calculations (see Appendix 4), if the % purity is ≥97%, the difference between that and 100% may be considered negligible for practical purposes and 100% purity may be used for standards calculation.

B $$10mls * \frac{1L}{1000mls} = 0.01L$$

C) $$\frac{225mg}{0.01L} = 22500mg / L$$

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2) Calculation as a Single Step.

$$\frac{0.2500gm}{10ml} * 0.90(purity) * \frac{1000mg}{1gm} * \frac{1000ml}{1L} = 22500mg / L$$

3)

$$FC = \frac{W}{FV} * P * \text{Conversion Factors}$$

where;

W = Weight of neat material (g)
FV = Final Volume (ml)
P = Purity (%/100)
FC = Final Concentration (mg/L = ppm)

On occasion, when preparing a standard from neat material, particularly a salt, the target analyte is only part of that salt (i.e. when preparing a chloride standard, for instance, one might use sodium chloride as the neat material). One conversion that would then be necessary is to convert the weight of the salt to the species of interest, in our example, chloride. In this case,

$$\text{Conversion Factor} = \frac{(\text{atomic weight of species of interest})(\text{\# of ions of interest in the molecule})}{(\text{molecular weight of the salt})}$$

**2. Intermediate and Working Standards (Standard Dilution)**

(FC)(FV) X 1000 = (AV) (PC)
where;
FC: Final Concentration(s) in standard desired. Units=µg/mL.
FV: Final volume of the prepared standard. Units=mL.
1000: Conversion factor from mL to µL
PC: Parent Concentration (standard normally containing high concentrations and is diluted to desired final concentration). Units = µg/mL.
AV: Aliquot Volume of parent standard required to achieve final concentrations desired. Units: µL (microliter).

a) Neats to Stocks

$$\frac{Purity * 1{,}000{,}000 * W}{FV} = FC$$

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where;

1,000,000 = Conversion factor from gram to microgram
W Weight used in standard prep (g)
FV Final Volume (ml)
FC µg/ml = ppm = mg/L
Purity = % Purity/100

For Example: 100% = purity of 1.0
86% = purity of 0.86

Although the iLIMS will handle any purity calculations (see Appendix 4), if the % purity is ≥97%, the difference between that and 100% may be considered negligible for practical purposes and 100% purity may be used for standards calculation.

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## Appendix 2

### Shelf Life Guidelines

**NOTE:** *IN NO CASE, will the Laucks' expirations date EXCEED the manufacturer's expiration date.
IN NO CASE, will a child solution have an expiration date that exceeds its parents.

| TYPE OF STANDARD | INORGANICS | ORGANICS EXTRACTIONS | ORGANICS INSTRUMENTATION[A] |
|---|---|---|---|
| Purchased Neat | 10 Years *[1] | 5 Years *[1] | 5 Years *[1] |
| Purchased Stock Solution | 12 Months *[2] | 12 Months *[2] | 12 Months *[2] |
| Prepared Stock Solution | 12 Months | 12 Months | 12 Months |
| Intermediate Solution | 3 Months | N/A | 6 Months |
| Working Solution | 2 weeks *[3] | 6 Months | 6 Months |
| Purchased Working Solution | | 6 Months | 6 Months |

* 1. Unless the manufacturers expiration date is less than the following, purchased neat standards shelf life will not exceed 10 years materials from the date of receipt for inorganics and 5 years from the date of receipt or 3 years from the date opened for organic materials, whichever is shorter.
*2. Unless manufacturers expiration date is less than the following, purchased stock solutions or intermediates shelf life will not exceed 1 year from the date opened.
* 3. Listed time is maximum. Specific shelf-life criteria are detailed in the individual SOPs.

NOTE: THIS IS A GENERAL PROTOCOL. WHERE POSSIBLE, VERIFY THE INTEGRITY OF THE WORKING STANDARD SOLUTION BY MEETING THE ACCEPTANCE CRITERIA SPECIFIED IN THE ANALYTICAL SOP FROM THE KNOWN TRUE VALUE WHEN ANALYZED AGAINST AN INDEPENDENT LABORATORY CONTROL STANDARD OR A CALIBRATION CURVE.

A. Volatiles:

| Method | Expiration Date |
|---|---|
| CLP OLM04.3 & SOM01.1 | Stock Standards: 6 months or sooner.<br>Gases & reactive compounds: 2 months<br>Secondary standards: 1 month or sooner for gases & reactive compounds, e.g. styrene<br>Other purgeables: 6 months or sooner<br>IS, surrogate & matrix spike: fresh spiking solution weekly or sooner.<br>Calibration standards: weekly or sooner. |

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| Method | Expiration Date |
|---|---|
| | Standard solutions stored in ampulated glass vials for 2 years from preparation date or shorter if recommended by manufacturer. Once opened, expiration dates above apply.<br>Aqueous standards: 24 hours at 4°C or 1 hour at room temperature; 12 hours if stored on autosampler. |
| SW 846<br>8260B | Stock Standards: 6 months or sooner<br>Gases: weekly if unstable or 6 months if prepared in nitrogen<br>Working solutions: check frequently for degradation or evaporation<br>Calibration standards are prepared daily |

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**Appendix 3**

**Bench Sheet Examples**

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Runlog, Logbook: 31104, 31105 (Be only)
Analyst: BA/JMP
Method: 6020
Instrument ID: ELAN ICPMS

CCV = STD 3
ISTD: ME-15-54-20 (50µL/10mL Sample)

| Seq | Sample ID | Analysis Date | Time | DF | Matrix | Data File | Prep Batch | Analyst Comments |
|---|---|---|---|---|---|---|---|---|
| 1. | Blank | 5/10/04 | 10:50:33 | 1 | WATER | 051004Be.raw | | |
| 2. | Standard 1 ME-15-55-19 (B) | 5/10/04 | 10:57:19 | 1 | WATER | 051004Be.raw | | 10µL/50mL |
| 3. | Standard 2 | 5/10/04 | 11:04:04 | 1 | WATER | 051004Be.raw | | 100µL/50mL |
| 4. | Standard 3 | 5/10/04 | 11:10:51 | 1 | WATER | 051004Be.raw | | 500µL/50mL |
| 5. | Standard 4 | 5/10/04 | 11:17:39 | 1 | WATER | 051004Be.raw | | 1000µL/50mL |
| 6. | Standard 5 | 5/10/04 | 11:24:27 | 1 | WATER | 051004Be.raw | | 2000µL/50mL |
| 7. | QC Std 7 | 5/10/04 | 11:31:16 | 1 | WATER | 051004Be.raw | | |
| 8. | ICV1 ME-15-55-10 | 5/10/04 | 11:44:19 | 1 | WATER | 051004Be.raw | | 600µL/50mL |
| 9. | ICB1 | 5/10/04 | 11:52:03 | 1 | WATER | 051004Be.raw | | |
| 10. | CRDL1 ME-15-52-17 | 5/10/04 | 11:59:47 | 1 | WATER | 051004Be.raw | | 250µL/50mL |
| 11. | ICS-A1 ME-15-34-7(A) | 5/10/04 | 12:06:58 | 1 | WATER | 051004Be.raw | | 250µL/50mL |
| 12. | ICS-AB1 (A)+(B) | 5/10/04 | 12:13:42 | 1 | WATER | 051004Be.raw | | 250µL(A)+200µL(B)/50mL |
| 13. | CCV1 | 5/10/04 | 12:20:13 | 1 | WATER | 051004Be.raw | | |
| 14. | CCB1 | 5/10/04 | 12:27:44 | 1 | WATER | 051004Be.raw | | |
| 15. | B050304MSS01 | 5/10/04 | 12:34:14 | 5 | SOIL | 051004Be.raw | | |
| 16. | 0404354-02 | 5/10/04 | 12:41:03 | 5 | SOIL | 051004Be.raw | | |
| 17. | 0404354-19 | 5/10/04 | 12:47:33 | 5 | SOIL | 051004Be.raw | | |
| 18. | S050504MSS01 | 5/10/04 | 12:54:04 | 5 | SOIL | 051004Be.raw | | |
| 19. | 0404380-11S | 5/10/04 | 13:00:36 | 5 | SOIL | 051004Be.raw | | |
| | 0404383-05S | 5/10/04 | 13:07:08 | 5 | SOIL | 051004Be.raw | | |
| 21. | B050604MSS03 | 5/10/04 | 13:13:55 | 5 | SOIL | 051004Be.raw | | |
| 22. | S050604MSS03 | 5/10/04 | 13:27:06 | 5 | SOIL | 051004Be.raw | | |
| 23. | 0404352-02 | 5/10/04 | 13:33:55 | 100 | SOIL | 051004Be.raw | | |
| 24. | 0404352-04 | 5/10/04 | 13:40:44 | 100 | SOIL | 051004Be.raw | | |
| 25. | CCV2 | 5/10/04 | 13:47:32 | 1 | WATER | 051004Be.raw | | |
| 26. | CCB2 | 5/10/04 | 13:54:18 | 1 | WATER | 051004Be.raw | | |
| 27. | 0404312-04 | 5/10/04 | 14:01:05 | 100 | SOIL | 051004Be.raw | | |
| 28. | 0404312-10 | 5/10/04 | 14:07:55 | 100 | SOIL | 051004Be.raw | | |
| 29. | 0404312-12 | 5/10/04 | 14:14:45 | 100 | SOIL | 051004Be.raw | | |
| 30. | 0404312-15 | 5/10/04 | 14:21:31 | 50 | SOIL | 051004Be.raw | | |
| 31. | 0404313-03 | 5/10/04 | 14:28:15 | 100 | SOIL | 051004Be.raw | | |
| 32. | 0404313-04 | 5/10/04 | 14:34:59 | 50 | SOIL | 051004Be.raw | | |
| 33. | 0404397-17 | 5/10/04 | 14:41:43 | 1 | WATER | 051004Be.raw | | |
| 34. | 0404397-17D | 5/10/04 | 14:48:28 | 1 | WATER | 051004Be.raw | | |
| 35. | 0404397-17L | 5/10/04 | 14:55:13 | 5 | WATER | 051004Be.raw | | |
| 36. | 0404397-17S | 5/10/04 | 15:01:59 | 1 | WATER | 051004Be.raw | | |
| 37. | CCV3 | 5/10/04 | 15:15:24 | 1 | WATER | 051004Be.raw | | |
| 38. | CCB3 | 5/10/04 | 15:22:09 | 1 | WATER | 051004Be.raw | | |
| 39. | 0404397-17A | 5/10/04 | 15:28:54 | 1 | WATER | 051004Be.raw | | |
| 40. | 0404397-18 | 5/10/04 | 15:35:42 | 1 | WATER | 051004Be.raw | | |
| 41. | 0404397-08 | 5/10/04 | 15:42:28 | 1 | WATER | 051004Be.raw | | |
| | 0404397-08D | 5/10/04 | 15:49:16 | 1 | WATER | 051004Be.raw | | |
| 43. | 0404397-08L | 5/10/04 | 15:56:03 | 5 | WATER | 051004Be.raw | | |
| 44. | 0404397-08S | 5/10/04 | 16:02:52 | 1 | WATER | 051004Be.raw | | |
| 45. | 0404397-08A | 5/10/04 | 16:09:40 | 1 | WATER | 051004Be.raw | | |
| 46. | 0404397-09 | 5/10/04 | 16:16:30 | 1 | WATER | 051004Be.raw | | |

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Laucks Testing Laboratories, Inc.
BATCH SUMMARY (for databatch - \\THOR\tek4\Felix.i\F51104syn.b )

| | Filename | Lab Sample ID | Client Sample ID | SDG | I.V. | D.F. | Inj. Date & Time |
|---|---|---|---|---|---|---|---|
| 1 | F5110402.D | STD01 50PPB | HPLC1620/1611 400X | | 0 | 1 | 11-MAY-2004 14:20 |
| 2 | F5110403.D | STD01 50PPB | HPLC1620/1611 400X | | 0 | 1 | 11-MAY-2004 14:57 |
| 3 | F5110404.D | STD02 100PPB | HPLC1620/1611 200X | | 0 | 1 | 11-MAY-2004 15:34 |
| 4 | F5110405.D | STD03 500PPB | HPLC1620/1611 40X | | 0 | 1 | 11-MAY-2004 16:11 |
| 5 | F5110406.D | STD04 1000PPB | HPLC1620/1611 20X | | 0 | 1 | 11-MAY-2004 16:48 |
| 6 | F5110407.D | STD05 5000PPB | HPLC1620/1611 4X | | 0 | 1 | 11-MAY-2004 17:25 |
| 7 | F5110408.D | IBLK 2000PPB | HPLC1-7-9 10X | | 0 | 1 | 11-MAY-2004 18:02 |
| 8 | F5110409.D | ICV 2000PPB | HPLC1-7-1 10X | | 0 | 1 | 11-MAY-2004 18:39 |
| 9 | F5110410.D | STD04 1000PPB | HPLC1-6-20 20X | | 0 | 1 | 11-MAY-2004 19:17 |
| 10 | F5110411.D | B042804HORSMS | B042804HORSMS | 31108 | 0 | 1 | 11-MAY-2004 19:54 |
| 11 | F5110412.D | 0404313-01 | 03SS1140002 | 31108 | 0 | 1 | 11-MAY-2004 20:31 |
| 12 | F5110413.D | 0404313-03 | 03SS0880002 | 31108 | 0 | 1 | 11-MAY-2004 21:08 |
| 13 | F5110414.D | 0404313-03DL | 03SS0880002DL | 31108 | 0 | 20 | 11-MAY-2004 21:45 |

I.V. = Inj.Vol. per Column  Batch Summary  Ver 2.0 9-5-2002

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**Appendix 4**

**"HOW TO" iLIMS Documents**

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# IT How-to Document

Date: 2004.05.25 Author: Mike Nelson Version: 1.0

How-to subject: Using the purity factor for gravimetric conversions in the iLIMS standards log

Operation steps:

1) In some cases you will want to prepare a standard using a pure material, but express the concentration in an alternative way. For instance, you can use pure sodium chloride to prepare a standard for which you want to know the chloride concentration. Using the purity factor in an iLIMS standard provides you with a way to make this conversion.

   Consider the above example, expressing the chloride equivalent of a sodium chloride standard solution. First, one needs to compute the percent composition of the targeted analyte:

   NaCl ---> Cl • •

   Na x 1 = 23 •

   Cl x 1 = 35.5

   • ---------- •

   58.5 <-- total

2) The percentage of chloride in sodium chloride is 100 * (35.5/58.5) = 60.7%.
3) Start the iLIMS standards log application and choose to add a new neat standard. When the main data entry screen is available, enter the computed gravimetric conversion factor, as above, in the 'Purity (%)' field of the form. Add the appropriate analyte(s), chloride in this case.
4) Use the NOTES section of this standard solution to fully document the information that you used to compute the purity. This will allow a reviewer to check your work.
5) Enter the special analyte, chloride in this case, as the analyte for this neat material.
6) Example 2, multiply-valent salts.

   Consider the computation of the nitrogen equivalent of $(NH_4)_2SO4$

   First compute the elemental composition:

   N = 14 x 2 = 28

   H = 1 x 8 = 8

   S = 32 x1 = 32

   O = 16 x 4 = 64

   Total = 132

   Percentage of nitrogen = 100 * (28/132) = 21.2%

   You would use this percentage composition in the purity for ammonium sulfate to determine the nitrogen equivalent of this standard.

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IT How-to Document

Date: 2004.04.30 Author: Della Wilson Version: 1.0

How-to subject: Handling Co-eluting and Degradation Compounds in the iLIMS

Operation steps:

Co-eluting Compounds

1. Compounds such as 3-methylphenol and 4-methylphenol co-elute on the GC/MS analysis appearing as one peak. The instrument software may be set up to identify it as either 3-methylphenol or 4-methylphenol, or as the combined 3&4-methylphenol compound. M-xylene and p-xylene also co-elute and would treated in the same manner as explained in this document.
2. In the Standards logs, the standard solutions with the co-eluting compounds should have the **co-eluting compounds listed as the sum of the individual concentrations**. i.e. If the solution of 3-methylphenol and 4-methylphenol was constructed with a purchased solution of 100ug/ml of 3-methylphenol and 100ug/ml of 4-methylphenol, the ILIMS purchased standard solution should list the analyte concentrations as 200ug/ml 3-methylphenol, 200ug/ml 4-methylphenol and 200ug/ml 3&4-methylphenol. The Manufacturer's individual concentrations can be recorded in the Standard's Notes section with the comment that these compounds co-elute.
3. The Test Determination may list any of the 3 analyte names: 3-methylphenol, 4-methylphenol or 3&4-methylphenol.
4. The instrument file may list any of the 3 analyte names: 3-methylphenol, 4-methylphenol or 3&4-methylphenol.
5. The Run Sequence's electronic importing of the instrument file will handle any of the 3 analyte names. If you are using the combined name of 3&4-methylphenol in the instrument file but the determination is reporting 3-methylphenol (and/or 4-methylphenol), you will have to identify that "3&4-methylphenol" is an alias for 3-methylphenol (and/or 4-methylphenol). You can identify alias names by using "Identify Analytes" located on the left side of the screen in run sequence. You only need to identify alias names once and the database will always look for that alternate name if it does not find the original name during electronic importing.

Degradation Compounds

1. Some compounds are not identified in an instrument analysis because they are unstable in the environment or the conditions under which they are analyzed cause them to degrade to different compound. i.e. N-nitrosodiphenylamine rapidly degrades to diphenylamine.
2. In the Standards logs, the standard solutions with the original compound should also have **the degradation compound listed with the same concentration as the**

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**original compound**. i.e. If a solution of 100ug/ml N-nitrosodiphenylamine was purchased, the ILIMS purchased standard solution should list the analyte concentrations as 100ug/ml N-nitrosodiphenylamine and 100ug/ml Diphenylamine. It should be recorded in the Standard's Notes section with the comment that N-Nitrosodiphenylamine degrades to Diphenylamine.

3. The Test Determination may list either N-Nitrosodiphenylamine or Diphenylamine per the client's request.
4. The instrument log may list either N-Nitrosodiphenylamine or Diphenylamine.
5. The Run Sequence's electronic importing of the instrument file will handle either analyte name. But you will have to identify that "N-nitrosodiphenylamine" is an alias for Diphenylamine and that "Diphenylamine" is an alias for N-nitrosodiphenylamine. You can identify alias names by using "Identify Analytes" located on the left side of the screen in run sequence. You only need to identify alias names once and the database will always look for that alternate name if it does not find the original name during electronic importing.

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# IT How-to Document

Date: 2004.05.18 Author: Della Wilson Version: 1.0

How-to subject: Converting Molarity or Normality Solutions to Concentration Units

Operation steps:

**Converting Molarity**

Molarity of Solution x Formula weight of the solution x 1000mg/gm = Conc. units of mg/L

Molarity: M = moles of solute (mol) / volume of solution (L)

Formula Weight: grams of solute (gm) / mole of solute (mol)

Examples:

1 M NaOH solution

$$1 \frac{mol}{L} \text{ NaOH} \times 40.00 \frac{gm}{mol} \text{ NaOH} \times 1000 \frac{mg}{gm} = 40{,}000 \frac{mg}{L}$$

1 M HCl solution

$$1 \frac{mol}{L} \text{ HCl} \times 36.46 \frac{gm}{mol} \text{ NaOH} \times 1000 \frac{mg}{gm} = 36{,}460 \frac{mg}{L}$$

3.5 M Sulfuric Acid solution

$$3.5 \frac{mol}{L} \text{ H2SO4} \times 98.07 \frac{gm}{mol} \text{ H2SO4} \times 1000 \frac{mg}{gm} = 343{,}245 \frac{mg}{L}$$

**Concentration of individual atoms in a Molar Solution**

Use the atomic weight of the individual atom instead of the formula weight of the compound.

Molarity of Solution x Atomic weight x 1000mg/gm = Concentration units of mg/L

Example:
Concentration of Calcium in a 0.01M solution of CaCO3

$$0.01 \frac{mol}{L} \text{ CaCO3} \times 40 \frac{gm}{mol} \text{ Calcium} \times 1000 \frac{mg}{gm} = 400 \frac{mg}{L}$$

**Converting Normality Solutions**

Normality is an integer multiple of Molarity and depends upon the equivalent number of hydrogen ions (H+) or electrons (e-) furnished or accepted. Normality/Equivalence = Molarity

First, convert from Normality to Molarity. Then apply the formula stated above to convert Molarity to concentration units.

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# IT How-to Document

Date: 2004.05.18 Author: Della Wilson Version: 1.0

How-to subject: Converting Molarity or Normality Solutions to Concentration Units

Examples:

1 N solution of NaOH

1 N NaOH = 1 equivalence, or accept 1 hydrogen ion

$$\frac{1\ N\ NaOH}{1} = 1\ M\ NaOH = 1\ \frac{mol}{L}\ NaOH$$

$$1\ \frac{mol}{L}\ NaOH \times 40.00\ \frac{gm}{mol}\ NaOH \times 1000\ \frac{mg}{gm} = 40{,}000\ \frac{mg}{L}$$

So, 1 N solution of NaOH has 40,000 mg/L of NaOH.

0.25 N solution of NaOH

0.25 N NaOH = 0.25 equivalence, or accept 0.25 hydrogen ions

$$\frac{0.25\ N\ NaOH}{0.25} = 0.25\ M\ NaOH = 0.25\ \frac{mol}{L}\ NaOH$$

$$0.25\ \frac{mol}{L}\ NaOH \times 40.00\ \frac{gm}{mol}\ NaOH \times 1000\ \frac{mg}{gm} = 10{,}000\ \frac{mg}{L}$$

So, 0.25 N solution of NaOH has 10,000 mg/L of NaOH.

1 N solution of H2SO4

1 N H2SO4 = 2 equivalence, or furnish 2 hydrogen ions

$$\frac{1\ N\ H2SO4}{2} = 0.5\ M\ H2SO4$$

$$0.5\ \frac{mol}{L}\ H2SO4 \times 98.07\ \frac{gm}{mol}\ H2SO4 \times 1000\ \frac{mg}{gm} = 49{,}035\ \frac{mg}{L}$$

0.0141N Mercuric Nitrate

0.0141 N Hg(NO3)2 = 2 equivalence, or furnish 2 electrons

$$\frac{0.0141\ N\ Hg(NO3)2}{2} = 0.00705\ M\ Hg(NO3)2$$

Formula weight for Mercuric Nitrate, Monohydrate is 342.62 gm/mol.

$$0.00705\ \frac{mol}{L}\ Hg(NO3)2 \times 342.62\ \frac{gm}{mol}\ Hg(NO3)2 \times 1000\ \frac{mg}{gm} = 2415\ \frac{mg}{L}$$

0.0141 N Silver Nitrate

0.0141 N AgNO3 = 1 equivalence, or furnish 1 electrons

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**IT How-to Document**

Date: 2004.05.18 Author: Della Wilson Version: 1.0

How-to subject: Converting Molarity or Normality Solutions to Concentration Units

$$\frac{0.0141\ N}{1}\ AgNO_3 = 0.0141\ M\ AgNO_3$$

Formula weight for Silver Nitrate is 169.87 gm/mol.

$$0.0141\ \frac{mol}{L}\ AgNO_3 \times 169.87\frac{gm}{mol}\ AgNO_3 \times 1000\ \frac{mg}{gm} = 2395\ \frac{mg}{L}$$

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# IT How-to Document

Date: 2003.06.13 Author: Mike Nelson Version: 1.0

How-to subject: Preparing standards with non-concentration units

Operation steps:

1) Log in to the iLIMS and start the standards log application.
2) Add a purchased solution to the log that has the analyte(s) you want with the exact same "concentration" and units as you want to have in your working solution. For instance, if you want to end up with a conductivity solution and 100umhos/cm then you must add a purchased solution that's expressed as 100 umhos/cm.
3) Save this solution.
4) Add an in-house standard solution that is made up using the purchased solution you just entered and having identical initial and final volumes. Save this solution. If you get errors when you try to calculate the final concentration then we may need to add additional bogus concentration units or conversion factors to cause the final concentration to be computed properly and for you to be able to get a report. If you get such errors then see a system administrator for help on adding such units.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1017

Title: **Internal Audit Procedures**

Revision history:

| Number | Date |
|---|---|
| 0 | 5/13/93 |
| 1 | 3/3/96 |
| 2 | 3/19/03 |
| 3 | 3/28/05 |

Written by: Harry Romberg, QA Officer Date: 4/21/05

Approved by: Kathy Kreps, Laboratory Director Date: 4/21/05

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this procedure is to provide instructions for planning, performing and reporting QA/QC audits within the laboratory.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the technique described.

1.2 Discussion

1.2.1 An audit of the facility is performed for the following reasons:

1.2.1.1 To determine that contractual and regulatory obligations are fulfilled and agency requirements met including but not limited to issues identified in external audits.

1.2.1.2 To determine that procedures and standards are being followed, and to insure good laboratory practice. These audits will include, but are not limited to the refrigeration unit temperatures, logbooks, balance calibrations, data, and standards traceability.

1.2.1.3 To establish that quality assurance objectives are met, including holding times, use of approved analytical methods, and stated objectives for precision and accuracy

1.2.1.4 To serve as a management tool to evaluate appropriateness of quality assurance policies.

1.2.1.5 To identify potential or actual deficiencies for the purposes of evaluating compliance with requirements and providing the means for correction.

1.2.1.6 To determine that records are prepared and maintained as required.

1.3 Documentation and Frequency

1.3.1 Documentation required is specified in the text and the frequency shall be as required by the QA Manager but at least one technical audit shall be performed annually for each department. This audit may take place in parts, with additional and more extensive audits being scheduled as deemed necessary.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP.

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1.4.1.1 **Audit** – A systematic evaluation to determine the conformance to quantitative and qualitative specifications of some operational function or activity.

1.4.1.2 **Auditor** - An individual who performs or assists in the performance of any part of an audit, including technical specialists.

1.4.1.3 **Corrective Action** – The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation. The action may be immediate and correct only the specific nonconformity or it may be long-term and correct an undesirable practice that could otherwise lead to additional nonconformities in order to prevent recurrence.

1.4.1.4 **Data Audit** – A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

1.4.1.5 **Deficiency** - Departure from approved procedures, program requirements, other applicable documents or good management practices that, if not corrected in a timely manner, could reasonably be expected to have a future adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **minor** discrepancy in the audit report.

1.4.1.6 **Discrepancy** - Departure from approved procedures, program requirements or other applicable documents that have or may have an adverse effect on the adequacy or effective implementation of the Laucks QA program. This includes findings and deficiencies found during the course of an audit.

1.4.1.7 **External Audit** – An evaluation of the laboratory conducted by an outside authority whether that be an agency or a client or a third entity in order to determine if the laboratory has the systems, capabilities and mechanisms to produce data in accordance with good laboratory practices and regulatory, contractual or other requirements.

1.4.1.8 **Finding** - Departure from approved procedures, program requirements, or other applicable documents that have, or in the immediate future could reasonably be expected to have, an adverse effect on the adequacy or effective implementation of the Laucks QA program. This would be ranked as a **critical or major** discrepancy in the audit report, depending upon severity.

1.4.1.9 **Internal Audit** – An evaluation conducted by the laboratory itself to determine if it is meeting its own quality criteria as well as meeting the requirements and objectives of other agencies or programs.

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1.4.1.10 **Lead Auditor** - An individual who is qualified to organize and direct an audit, report audit findings, and evaluate proposed corrective actions.

1.4.1.11 **Performance Audit** – The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

1.4.1.12 **Recommendation** - An observation or advise given to enhance current practices by any individual or department of the Laucks QA program. This would be ranked as a **recommended** item in the audit report.

1.4.1.13 **SOP** - Standard Operating Procedure – A written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks.

1.4.1.14 **Systems Audit** – A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management and reporting aspects of a total measurement system.

1.4.1.15 **QA** - Quality Assurance – An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

1.4.1.16 **QC** - Quality Control – The overall system of technical activities whose purpose is to measure and control quality of a product or service so that it meets the needs of users.

## 2. Responsibilities

2.1 QA Personnel and Audit Team

2.1.1 It is the responsibility of QA personnel, the auditor and audit leader to perform an audit according to this SOP and complete all documentation required for review.

2.1.2 Audits and reports are to be generated by personnel in the laboratory who have demonstrated the ability to evaluate processes in the laboratory with emphasis on Quality Control and Quality Assurance.

2.1.3 QA Managers are responsible for the following:

- Approving each detailed audit plan

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- Concurring with the adequacy of each audit report
- Issuing the audit report
- Tracking audit status through final closeout

2.1.4 If an audit team is used, the following responsibilities fall upon the Audit Team Leader. If an audit team is not used, they fall upon the QA Manager.

- Developing the detailed audit plan
- Conducting pre-audit and post-audit conferences
- Supervising the conduct of the audit
- Preparing and signing the audit report
- Final review and sign-off of each Audit Finding Report may be performed by either the QA Manager, Lab Director or department supervisor or designee as appropriate.

2.2 Supervisors and Department Personnel

- Providing reasonable and timely access to personnel, facilities, and records, as required to support the audit process
- Providing timely and adequate response to audit reports, including determination and implementation of corrective actions, as required.
- Respond to audit findings using the provided forms including providing a planned date implementation of corrective actions
- Develop and implement planned corrective actions
- If findings in one audited area exist in other areas, implement appropriate corrective actions in those areas as well.
- Verifying initial implementation of corrective action for deficiencies in their areas, if applicable.

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## 3. Safety precautions

3.1 Safety Precautions

3.1.1 Auditors must adhere to the general laboratory health and safety policies during the course of the audit.

3.1.2 Protective eyewear must be worn in all applicable locations at all times during the course of the audit.

## 4. Operation Procedures

4.1 General

4.1.1 Audit personnel may be selected and assigned audit responsibilities commensurate with their training and expertise and the special nature of the activities to be audited.

4.1.2 Audit personnel are independent of any direct responsibility for performance of any activity which they will audit. Persons having direct responsibility for performance of the activities are not involved in the audit team but supervisors may provide guidance regarding items which they feel should be observed by the audit team.

4.2 Audit Planning

4.2.1 The QA Manager, or designee shall develop an audit plan which shall be the basis for the audit. The plan will involve checklists common to the specific area to be audited but it may also be free-form in order to enable the auditor(s) to ask questions or address concerns that arise during the audit that were not addressed in the checklist.

4.2.2 In addition, specific areas of concern may be suggested from previous external audits and from supervisory or other personnel who have reason to think a specific topic should be addressed either because they think it may not be properly managed or because review is deemed appropriate due to recent changes or past performance issues.

4.2.3 Though checklists are used to structure an audit, they may be modified from one audit to the next at the discretion of the auditor. Some areas may be addressed in one audit and then the focus shifted to another area in a subsequent audit so the forms may be modified from one audit to the next.

4.2.4 It may also be determined during the audit that a specific checklist does not address the area in the way that the auditor would like. At the discretion of the auditors, it may be determined that a different focus is appropriate and the auditors are free to improvise in order to pursue a particular topic not previously addressed in the plan/checklist.

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4.2.5 Audits are scheduled in a manner to provide coverage and coordination with ongoing QA program activities.

4.2.6 Audits are scheduled at a frequency commensurate with the status and importance of the activity. Within the audit program, each department of the laboratory and each element of the Laucks-QA program is audited, at a minimum, at least once annually. A schedule is provided in appendix III but areas can be switched at the discretion of QA, particularly if one area is suspected to be under performing or a critical error has occurred that suggests closer scrutiny is in order and requires a review prior to the scheduled date. Items may also be added to this schedule at the discretion of QA for any reason.

4.2.7 The QA Manager notifies the audited department prior to the audit to provide the subject and scope of the audit, audit schedule, and audit team members, if applicable

4.3 Audit Performance

4.3.1 The QA Manager and (when required) the appointed audit team members shall proceed through the audit checklist recording evidence of compliance, discrepancies, or recommendations.

4.3.2 During the audit, the Auditor(s) shall use their best judgment to determine if there is a need to audit at a greater depth than the checklist indicates. If this is the case, the checklist shall be modified accordingly.

4.3.3 Objective evidence is examined, and essential information is recorded, such as the identification of specific evidence examined, specific details of discrepancies or adverse conditions, and applicable references.

4.3.4 The QA Manager shall identify each finding, deficiency, or recommendation in a QA audit report. Findings, deficiencies and recommendations will be listed by department and sequentially numbered in the QA audit report.

4.4 Audit Report

4.4.1 The QA Manager or designee shall prepare an audit report which should address the following:

- Date and location (Laucks-department) of the audit.
- Purpose and scope of the audit.
- Audit team members (when applicable).

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- Description of items, including the rank. The description of the items must be in sufficient detail to enable investigation, evaluation, and correction of the finding. The report may also include the area affected (Table 1) and Finding Type (Table 2).

- Due date for completion of corrective action plans.

- Means for verifying completion of corrective action.

4.5 Audit Closure and Follow-Up

4.5.1 The appropriate Laucks Management (departmental supervisors, laboratory manager, etc.) shall investigate the reported finding, deficiency or recommendation and do the following:

4.5.2 Determine the actions required to correct the discrepancy.

4.5.3 Evaluate each discrepancy to determine the root cause of the problem and any generic implications.

4.5.4 Determine the corrective action required to correct the discrepancy and to prevent recurrence.

4.5.5 Document corrective action and indicate corrective action commitment date.

4.5.6 Sign, date, and return the completed form to the QA Manager within the assigned time frame given in the audit report.

4.5.7 The QA Manager shall evaluate each discrepancy/recommendation response. Inadequate or indeterminate responses shall be returned for reexamination of the problem and revised corrective action.

4.5.8 The QA Manager shall verify the corrective action, as stated in the response, and make sure it has been implemented and accomplished as scheduled.

4.5.9 After verification of corrective action, the QA Manager shall issue a closure memo or letter stating that all corrective action has been completed and the audit is closed.

4.5.10 If a stalemate is reached concerning either the validity or resolution of an audit finding, affected personnel escalate the concern to the appropriate level of management to effect a resolution.

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4.6 Records

4.6.1 The QA Manager shall ensure that the following audit documentation is maintained on file:

- Completed audit checklist.
- Audit Report (includes findings, deficiencies and recommendations).
- Corrective Action (response to discrepancies).
- Records pertaining to the completion of corrective action.

4.7 Audit Discrepancy Tracking

4.7.1 Audit discrepancies will be categorized to facilitate tracking and trending of recurrent problems. The categories are as follows:

- Logbook Maintenance
- Document Control Procedures
- QC Procedures
- Standard Operating/Quality Assurance Procedure
- Analytical Method
- Purchasing/Procurement Document Control
- Standards Preparation/Documentation
- Safety/Reagent Labeling or Storage
- Training/Records
- Good Laboratory Practices
- Other

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4.7.2 Explanations of Categories Listed Above

4.7.2.1 Logbook maintenance findings include but are not limited to the following: logbooks not being maintained in accordance with Laucks policy, improper entries into logbooks, improper error corrections, logbooks not being kept up to date.

4.7.2.2 Document Control Procedure findings include but are not limited to the following: documents being maintained in such a way that is non-complaint with Laucks document control procedures (this includes archives, SOPs, QAPs, Chemical Hygiene Plan, OOCEs, and forms), records being stored in work areas for longer than 6 months, improper handling of controlled procedures.

4.7.2.3 QC procedure finding include but are not limited to the following: temperatures of ovens and refrigeration units not being monitored in accordance with procedures, balances and pipettes not being verified as required.

4.7.2.4 Standard Operating Procedure and Quality Assurance procedure findings include any case where a procedure has not been followed in full and has not been documented on an OOCE.

4.7.2.5 Analytical methods findings involve cases where the approved and required analytical method has not been followed to the full extent and there is no documentation that communicates this.

4.7.2.6 Purchasing and procurement document control findings involve instances where the appropriate procedures have not been followed in full. This type of finding includes but is not limited to the following: un-approved changes to purchase requisitions, lack of certification documentation, etc.

4.7.2.7 Findings for standards preparation and standards documentation include but are not limited to the following circumstances: improperly prepared standards, improperly documented standard preparation, inadequate verification documentation, lack of documentation when procedures are not followed in full.

4.7.2.8 Safety and reagent/chemical labeling findings involve any deviation from approved safety and waste procedures and the chemical hygiene plan.

4.7.2.9 Training and training records findings involve lack of training records, and personnel performing analysis without appropriate qualification documentation.

4.7.2.10 Good Laboratory Practice findings involve significant figures, temperature monitoring, calibration techniques and other associated activities involved with safe and accurate laboratory practices. This finding is often used to classify

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findings that do not fit cleanly into some of the standard categories and, due to uniqueness or infrequency, do not warrant creation of an additional category.

4.7.3 QA may at any time add to the categories and specific discrepancies for areas that appear to require this level of detail. No matter what the category or discrepancy type, however, it will be accompanied by a narrative explanation of the specific observation.

## 5. References

Laucks Quality Assurance Plan

Applicable SOPs

Audit Database Tables

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**Appendix I**

**Audit Finding Report Form**

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## Audit Finding Report

| | |
|---|---|
| Audit Number: | Finding Number |
| Facility: | Audit Date: |
| Auditing Body: | Audit Type: |
| Lead Auditor: | Affected Area: |

Related Findings:

Finding Rank: Repeat Finding?:

Finding:

Corrective Action Response:

| | |
|---|---|
| Opened By: | Date Opened: |
| Response By: | Response Date: |
| Corrective Action By: | Scheduled Completion Date: |
| Verified By: | Date Verified: |

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**Appendix II**

**Finding Types**

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| ID of Finding Type | Finding Type |
|---|---|
| BA1 | Balance - Not Certified Annually |
| BA2 | Balance - Not Checked Daily With Class S Weights or as used |
| BA3 | Balance - Weights Not Certified Annually |
| BA4 | Balances - Weights used for calibration do not correspond to weights used for analysis |
| CA1 | Corrective Action - Procedures Not Developed |
| CA2 | Corrective Action - NVC Not Being Tracked |
| CH1 | Control Charts not maintained |
| CH2 | Control Charts - Corrective actions not taken or documented |
| DA1 | Data Review-Required information missing or incomplete |
| DA2 | Data Review-calculations incorrect or could not be verified |
| DA3 | Data Review-data was presented that was not required |
| DA4 | Data Review-data package was not assembled in the correct order or was difficult to interpret |
| DL1 | Documentation/Logbooks - Error and Corrections not be documented correctly |
| DL2 | Documentation/Logbooks - incomplete columns, not properly bound |
| DL3 | Documentation/Logbooks - Not Maintained or used |
| DL4 | Documentation/Logbooks - Inadequate Review |
| DL5 | Documentation/Logbook inaccurate, inappropriate, or incomplete |
| DR1 | Data Review - Not Being Performed |
| DR2 | Data Review - Not Being Documented |
| DR3 | Data Review - No SOP |
| DR4 | Data Review - No QC Decision Matrix Available |
| EC1 | Electronic Backup - Not Being Performed |
| EC2 | Electronic Backup - Not Inventoried For Retrieval |
| GL1 | Good lab practice - misc. GLP items |
| MD1 | Methods - No SOP/crib sheet available at time of audit |
| MD2 | Methods - SOP/Crib sheet in use not current controlled version |
| MD3 | Method- controlled SOP/Crib sheet is not being followed or doesn't match current practice |
| MD4 | Methods - The controlled SOP is Non-compliant with the referenced published method |
| MD5 | Methods - SOP/Crib sheets in use & not controlled, meaning draft, handwritten or unapproved SOPs in use |
| PE1 | Performance Evaluation Samples - Results are outside warning limits, check for error |

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| ID of Finding Type | Finding Type |
|---|---|
| PE2 | Performance Evaluation Samples - Results are outside control limits, not acceptable |
| PE3 | Performance Evaluation Samples - Results included misidentified compounds, not acceptable |
| QA1 | QA - QAP/SOP Document Control Not in Place or Used |
| QA2 | QA - Precision and Accuracy Data Not Current |
| QA3 | QA - MDL/IDL Not Current |
| QA4 | QA - QC Limits Not Determined or Maintained |
| QA5 | QA - Control Charts Not Developed or Maintained |
| QP1 | QA Plan - No QAP Available |
| QP2 | QA Plan - Outdated And Needs Revision |
| QP3 | QA Plan - Has Major Discrepancies With SOPs or practices of the day |
| QP4 | QAP- missing information for a particular program or QAP requirement |
| RC1 | Records Control - Logbooks Not Controlled |
| RC2 | Records Control - Filing not maintained per SOP |
| RC3 | Records Control - No SOPs to describe System |
| RC4 | Records Control - Not mentioned in QAP |
| RC5 | Records Control - Archiving inadequate |
| SC1 | Sample Control - Building not secured |
| SC2 | Sample Control - COC not established or maintained per client requirements |
| SC3 | Sample Control - Temp/pH not monitored for all regulatory samples |
| SC4 | Sample Control - Samples storage area not kept in locked |
| SF1 | Safety - No SOP |
| SF2 | Safety - Not Adhering to SOP or Chemical Hygiene Plan |
| SF3 | Safety - Not Adhering to Good Lab Safety Practices |
| SO1 | SOP- The SOP book did not contain the SOPs listed in the current Table of Contents(rev. or missing) |
| SO2 | SOP - The database had incorrect SOP information |
| SO3 | SOP - SOPs not reviewed or revised annually |
| ST1 | Stds/Reagents - No SOPs for preparation |
| ST2 | Stds/Rgnts - Prep record inadeq./or not traceable |
| ST3 | Stds/Reagents - Expiration Date Misused |
| ST4 | Stds/Reagents - Not Labeled Properly in the laboratory |
| ST5 | Standard/reagent certificates of authenticity not traceable or properly archived |
| ST6 | Stds/Reagents - not properly stored |
| SW1 | Software - Not Verified and Documented |

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| ID of Finding Type | Finding Type |
|---|---|
| TH1 | Thermometer - NIST Not Available |
| TH2 | Thermometer - NIST Not Evaluated Annually |
| TH3 | Thermometers - Not Calibrated Annually |
| TH4 | Thermometers - Correction Factor Not Applied or misapplied |
| TH5 | Thermometers - Temp. Not Recorded Daily or As Used |
| TH6 | Thermometer records incomplete |
| TR1 | Training - No Formal Program or Documentation |
| TR2 | Training - Incomplete Forms (e.g. Proficiency, Hrs) |
| TR3 | Training - Not Maintained Consistently, training files missing current info. |

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**Appendix III**

**Audit Schedule**

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**Laucks Testing Laboratories**

Internal Audit Schedule

| Department | Date |
|---|---|
| Extractions | January |
| GC/MS Volatiles | February |
| GC/MS Semivolatiles | March |
| GC Gasoline/Volatiles | April |
| GC Extractable Fuels | May |
| GC/ECD (Pesticides/Herbicides) | June |
| HPLC (Explosives) | July |
| Sample Receiving | August |
| Balances and Balance Logbooks | September |
| DI Water Systems and Logbook | September |
| Conventional Chemistry (3 areas minimum) & Hg | October |
| Metals | November |
| Metals Digestion | December |

Areas can be switched at the discretion of QA, particularly if one area is suspected to be underperforming or a critical error has occurred that suggests closer scrutiny is in order and requires a review prior to the scheduled date. Items may also be added to this schedule at the discretion of QA for any reason.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1018

Title: **Overview of Review and Approval Practices for Validatable Data Packages**

Revision history:

| Number | Date |
|---|---|
| 0 | 6/17/96 |
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Written by: Harry Romberg Date: 6/13/07
Harry Romberg, Quality Assurance Officer

Approved by: Hugh Prentice Date: 6/13/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Description

1.1.1 This SOP is intended to provide an overview and general organization of data review practices employed for validatable packages. The actual data review processes and check lists specific to those types of analyses are covered in specific SOPs. A schematic diagram of the general review process is provided in Appendix I.

1.1.2 Validatable packages are often similar to the Contract Laboratory Program (CLP) presentation, although the actual analyses themselves and the applicable quality control (QC) may be from SW 846 or other references. If such is the case, the CLP format would be modified to meet the requirements of the referenced methodology. However, the overall review process remains the same.

1.1.3 AFCEE or DoD forms and reporting flagging styles are also sometimes required and may be substituted for the more common CLP style.

1.1.4 In-house (non-validatable) data packages receive much of the same review in that data and calculations are checked and they need to meet most of the same criteria but, since the same level of documentation is generally not required, higher level review may not be as thorough and does not follow the same process. It is not the intent of this SOP to outline the process for these data.

1.1.5 Due to the nature of data production and assembly of packages for their respective departments, the processes for the Inorganics area and the Organics area are different. This SOP will try to outline the practices in both areas of the laboratory.

1.1.6 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described function.

## 2. Equipment List

2.1 Equipment

2.1.1 Data package or the portion of the data package to be validated

2.1.2 SOPs, including associated checklists, for the validation of the analyses of interest

2.1.3 Reference methods (SW 846, etc.) and programmatic QA Plans (i.e. AFCEE and DoD)

2.1.4 Access to computer programs, etc. which may be required in order to complete the review process, including the actual report narrative.

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## 3. Definitions

3.1.1 Validatable Data Package – A complete, self-contained, standardized and organized data package that has adequate information for a reviewer to reconstruct the analytical sequence, calibration and quality control and to verify both the calculations and how well the method functioned for that particular data set.

3.1.2 SOPs – Standard Operating Procedures – written documents that describe a method or operation in adequate detail such that another qualified individual could follow the outlined procedures with reasonable likelihood of arriving at approximately the same result as another analyst following the same procedure. SOPs contain the steps that the individual must undertake, the sequence of events (analytical sequence) the expected quality control requirements and actions, and assorted other information necessary to repeat the procedure.

## 4. Responsibilities

4.1 Analyst

4.1.1 It is the responsibility of the analyst to provide the first level of data review and to ensure that all criteria have been met or their failure addressed prior to releasing the data for the next level of review. The analyst may only be the first level of review but is the most important in ensuring that the reported values reflect what was actually present in the samples. It is particularly important that the analyst be proactive in determining any actions that need to be taken in order that they may be completed within the holding time for that analysis and within the turnaround time required by the client.

- The analyst must ensure that the instrument was functioning properly at the time of analysis.
- The analyst will ensure that all data comply with the method and project-specific requirements and that any deviations or failures to meet criteria and corrective actions taken or determined to be unnecessary are documented in the project file. This will include the generation of the QC Outliers Report from the iLIMS and the initiation of a data review checklist.
- The analyst must check to see that all calibration criteria were met.
- The analyst must review all quality control data and ensure that criteria were either met or corrective action taken. This action may vary anywhere from simple narration in the report to re-analysis of the sample set, depending upon the QC failure and the method requirements.

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- The analyst will review the final data to see that they make sense, that is, the values determined are reasonable, do not disagree with other information the analyst may be aware of, and that the calculated values appear to agree with the raw data.
- The analyst will consult with their supervisor and/or QA to resolve discrepancies with which they are unsure how to proceed or for which supervisory approval is required.
- The analyst will either transcribe the data into the iLIMS either manually or electronically or will pass data to the person responsible for transcription in a format which can be easily interpreted.
- The analyst will assemble the data package suitable for the next step in the review and approval process. For Organics, this includes the complete assembly of all analytical runs and QC in the approved format designated in the analytical SOP. For Inorganics, although the analyst serves as the first level of review, the full final data package is usually created at the next level.

4.2 Peer or Secondary Review

4.2.1 In the conventional chemistries area, the data is first reviewed by a peer analyst (calculations, QC, etc.) and is then passed along to the data administration assistant. In the metals area, the analyst-reviewed data is passed directly to the data administration assistant.

4.2.2 In the Inorganics area (conventional chemistry), the complete data package is assembled by a data administration assistant. In the Inorganics area (metals), the complete data package is put together by a lead or manager. This assistant/lead/manager reviews the package to make sure all of the forms and QC are present and that the appropriate limits are used. If irregularities are noted, they are brought to the attention of the analyst or the supervisor for resolution. As the Organics analyst assembles their own data, both steps 4.2.1 and 4.2.2 are omitted in that area. The completed package goes directly to the supervisor or designee who does both the peer and supervisory review.

4.2.3 Data must receive a second level of review from a peer analyst. This analyst should be a person who is familiar with and capable of performing the analysis themselves. If there is no peer analyst available because the analyst in question is the only one experienced with the analysis or for other critical reasons, another qualified individual may substitute for the peer analyst. This person must still be familiar with all aspects of the calculations being performed and the relationships between data and performance of the method in order that the review can be properly conducted. In the conventional chemistry area, this review is performed by an experienced analyst or lead. In the metals area, this function is performed by the supervisor or designate after the package has been assembled. In Organics, supervisory staff perform this function and the final review outlined in 4.3. The peer analyst reviewer must:

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- Check 100% of the manual entries for transcription errors
- Check 100% of manual calculations for accuracy
- Spot check at least 10% of computer calculations to verify program validity
- Check for completeness of raw data or supporting materials
- Confirm spectral assignments and identification of TICs
- Check for appropriate use of significant figures and rounding
- Check reported values for dilutions
- Check for compliance with Method and project-specific requirements
- Check for reasonableness of data
- Consult with the analyst, supervisor and/or QA to ask questions or resolve discrepancies with which they are unsure how to proceed

4.3 Supervisor

4.3.1 The responsible supervisor or a designated alternate for the area in which the analysis is conducted must provide a technical review of the reported data. This level of review need not be as detailed as the peer review (except in the organics department where it is performed at the supervisory level) but must include:

- Checking for reasonableness and sensibility of the reported data, including interrelationships between different data.
- Checking for completeness of the reported information
- Checking for compliance required QC practices including those specified in the Method and those that are project-specific.
- Checking for descriptions of deviations from Method and project-specific QC requirements
- Checking the information in the report narrative for sensibility
- Questioning the analyst or peer reviewer in resolving discrepancies or lack of clarity that may arise and consulting with QA and/or the Lab Director when discrepancies arise with which they are uncertain how to proceed.

4.4 QA Review

4.4.1 QA cursorily reviews most data and periodically, in conducting data audits, reviews select packages more thoroughly. The cursory reviews are generally performed just prior to

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release of the data. In depth reviews almost always occur after release of the data and are intended more for a review and assessment of laboratory data and processes rather than an assurance of performance on that particular data package. QA may also choose to designate another qualified individual to perform any of these levels of review. Should quality issues arise that have a critical negative impact on the package being thoroughly reviewed, however, QA may call for more specific corrective action. QA may choose to go into any depth in review of data packages, but in general, most reviews will consist of:

- Checking for compliance with required QC practices
- Checking for reasonableness and sensibility of reported data, including data interrelationships
- Checking for deviations from method or project specific QC requirements
- Checking for compliance with SOPs (periodically)

4.4.2 QA will resolve discrepancies with the supervisor, analyst, or pertinent individual. Some issues may involve corrective actions that require a simple correction while others may require a complete change in routine practices. Documentation of these actions is detailed in the Corrective Actions SOP.

4.4.3 QA will usually approve and sign data package narratives.

4.5 Project Manager

4.5.1 Project managers do not perform technical reviews but do review case narratives to ensure compliance with contractual agreements. Their responsibilities include:

- Reviewing to ensure that the client requested methodology was used and referenced
- Reviewing and commenting on sample receipt issues
- Ensuring that sample entry comments were incorporated, and that concerns that were raised during the course of analysis which required client communication and decisions have been incorporated.
- Reviewing and signing project narratives.
- Reviewing the billing to ensure that the proper invoicing has occurred in conjunction with contractual agreement.
- Reviewing or overseeing review of electronic disk deliverables (EDDs).

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4.6 Management

4.6.1 Senior management reviews case narratives and other components of data packages, should they find cause. They may be responsible for approving the release (signing) of some report narratives or may substitute for other reviewers where appropriate.

## 5. Operation Procedures

5.1 General

5.1.1 The processes described below are general. Specific QC and practices, including most of the corrective actions resulting from QC failures are generally described in the appropriate SOPs. The specifics of the review process for individual analyses are specified in the respective data review SOPs along with their associated checklists.

5.1.2 The duties of individuals responsible for various levels of review are specified in the Responsibilities section of this SOP. It is the responsibility of each reviewer to be familiar with this SOP and those specific to their function.

5.1.3 Due to the nature of data production and assembly of packages for their respective departments, the processes for the Inorganics area and the Organics area are different. This section will try to outline the practices in both areas of the laboratory.

5.2 Corrective Action

5.2.1 Corrective action may be required at any point in the review process. This may include anything from simple recalculation or transcription of a result to complete reanalysis depending upon the circumstance. Many corrective actions are outlined in the applicable SOPs. However, anyone in the review process can either prompt corrective action or, in consultation with any of the others, determine that various actions are either necessary or unnecessary. Corrective actions are individualized to the procedure and circumstance and may be determined to be unnecessary if there is adequate information available to support the reported result. Likewise, if circumstances arise such that corrective action does not provide for improved validity of the data, such as reanalysis well after a holding time has expired, it may be determined that further action is pointless. No one in the process should pass along information for which there are unresolved issues. At a minimum, any problems, resolved or unresolved, require narration and/or notes in the data file.

5.3 Analyst

5.3.1 The analyst must be cognizant of the entire analytical process and document anything out-of-the-ordinary that goes on during the analysis. This may include on-the-spot corrective action, such as dilution and re-analysis. The analyst must also review the data during the

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production of final results to ensure that all criteria are met and that all appropriate commentary regarding the analysis and any extraordinary steps are clearly noted.

5.3.2 The analyst will then assemble the final data package according to the SOP and submit the data for review to the secondary reviewer. The work of the analyst is the most critical in the review process as this ensures the timely processing of the samples in order to meet holding and turnaround times.

5.3.3 When the analyst completes the data package, he/she will pass all of the associated materials along to the second reviewer.

5.4 Peer or Secondary Review

5.4.1 The secondary review will usually include use of the checklists associated with the data review SOPs. If in doubt, the secondary reviewer will ask the analyst for further information and not just pass along problems to the next level. In consultation with the supervisor or QA, data may be returned to the analyst for corrective action.

5.4.2 The secondary reviewer will pass the data and checklist along to the supervisor

5.5 Supervisor

5.5.1 The area supervisor or designate will perform the functions outlined under the Responsibilities section, paying special attention to data review checklist items which do not meet method specifications. The supervisor may determine that corrective actions are necessary in the pursuit of data of adequate quality or may consult with QA where the optimal practice is questionable. The supervisor should ensure that corrective actions are all completed and all report commentary is sound prior to submitting the data to the reports department.

5.6 Reporting

5.6.1 The reporting group assembles the respective data packages but bears no responsibility for review other than to ensure that all of the analyses are present in the package, that everyone has input their respective commentary into the report narrative, that all narrative comments have been printed and the appropriate parts of the data package have been assembled. This aspect is detailed in an SOP designed for that purpose.

5.7 Quality Assurance

5.7.1 QA performs cursory reviews of most narratives and data packages before release. QA may call for corrective action at any level should problems be observed which have not been dealt with in an appropriate manner prior to this late stage of reporting.

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Responsibility to spot and have errors corrected, however, must not be left up to QA if they are spotted earlier or the analysis and reporting of results will almost certainly be delayed and may even be compromised if reanalysis is required and the delay does not allow them to be completed within holding time.

5.7.2 QA or an approved designee will also perform a more thorough review of select data packages, the scope of which is at the discretion of QA and is not addressed in this SOP. Such review will be more detailed, however, and corrective actions may result which will impact the immediate data or, more likely, affect the processes involved in collecting, reviewing, or reporting data in general.

5.8 Project Management

5.8.1 Project managers review and sign project narratives. They will review only to ensure that the client requested methodology was used and referenced, that sample entry comments were incorporated, and that concerns that were raised during the course of analysis which required client communication and decisions have been incorporated. They must also review the billing to ensure that the proper invoicing has occurred in conjunction with contractual agreement. They may perform these tasks either before or after QA review.

5.8.2 Project managers are also responsible for the review of the electronic disk deliverables (EDDs), although they may assign the actual review to other capable individuals. Where there are data checker tools available to perform a particular review, they are used with minimal additional review. Where no such tools exist, the EDD is manually 100% checked for conformance with the hardcopy result forms (Forms 1). Additionally, about 20% of QC results are also checked, although where flags exist, QC results are 100% checked.

5.9 Management

5.9.1 Management may review and release (sign) narratives and usually will review in-house reports.

## 6. Reports and Documentation

6.1 Data Review and Signatures

6.1.1 Quality Control (QC) Outlier Reports generated from the iLIMS

6.1.2 Data review forms are provided in individual data review SOPs.

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6.1.3 Analyst/reviewer signatures occur on organics cover pages. Inorganics review signatures occur on data cover pages and supervisor signatures are included on both metals and conventional chemistry packages.

6.1.4 Management signatures appear on all final reports.

## 7. References

*National Environmental Laboratory Accreditation Program (NELAP)*, 2003 Standard

*Department of Defense Quality Systems Manual*, Final Version 3, March 2005

*Air Force Center for Environmental Excellence, Quality Assurance Project Plan (QAPP)*, Final Version 4.0.02, May 2006

Laucks SOPs

| | |
|---|---|
| LTL-7001 | Metals Data Review |
| LTL-8001 | GC Hydrocarbons Data Review |
| LTL-8002 | GC Pesticides/PCBs Data Review |
| LTL-8003 | GC Herbicides Data Review |
| LTL-8004 | GC Volatiles Data Review |
| LTL-8005 | GC Gas/BTEX Data Review |
| LTL-8203 | GC/MS Data Review |
| LTL-8302 | HPLC Ordnance 8330 Data Review |
| LTL-9001 | Inorganic Conventionals Data Review |

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**Appendix I**

**Data Review Schematic**

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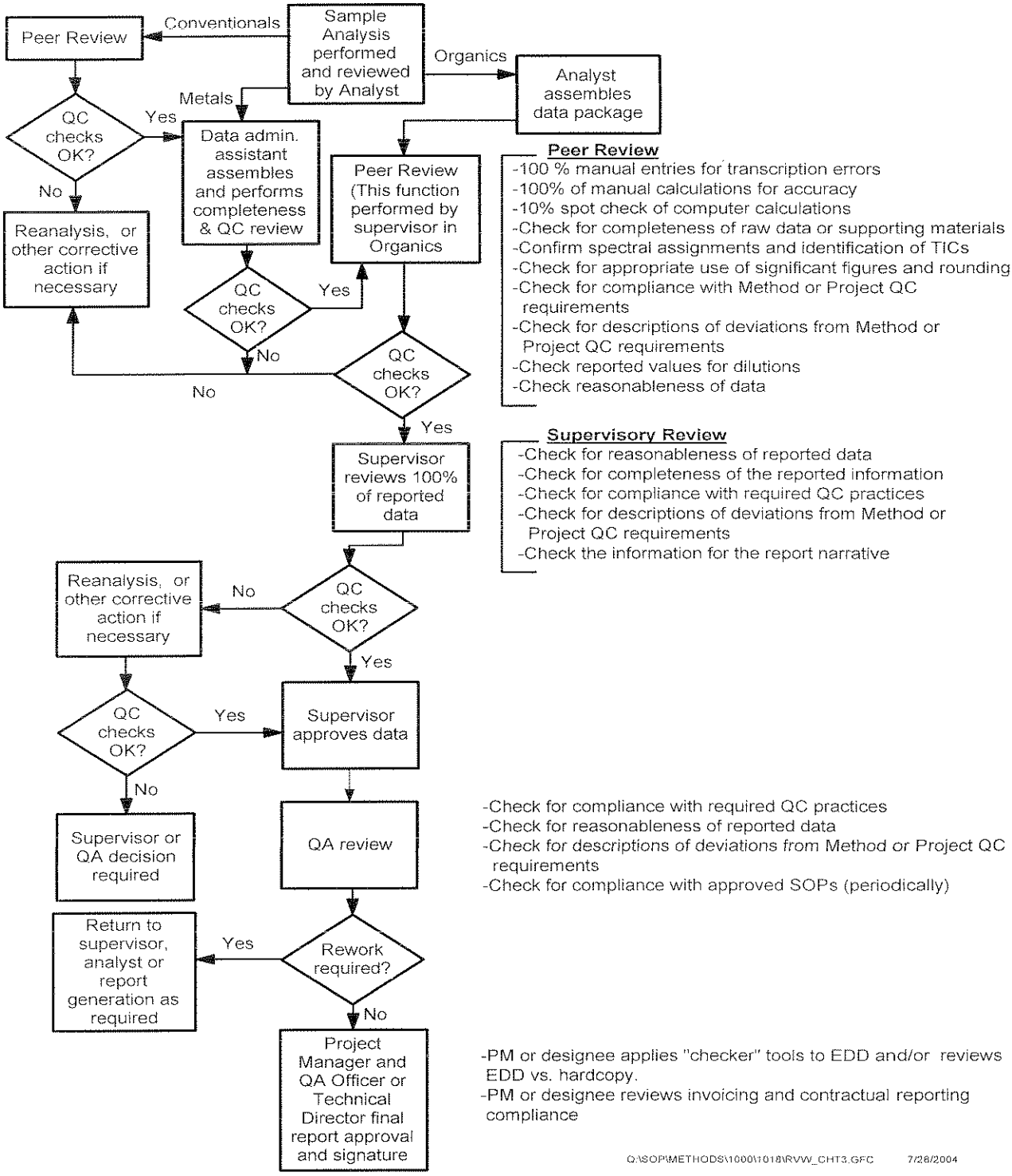
## Data Review and Approval



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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1019

Title: **Controlling, Maintaining, and Monitoring Laboratory Logbooks**

Revision history:

| Number | Date |
|---|---|
| 0 | 4/11/96 |
| 1 | 5/13/98 |
| 2 | 11/17/00 |
| 3 | 7/19/04 |
| 4 | 11/30/05 |

Written by: Harry Romberg, QA Officer Date: 11/30/05

Approved by: Kathy Kreps, Laboratory Director Date: 12/13/05

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## 1. Introduction and Scope

1.1 Scope

1.1.1 The maintenance of logbooks is essential to monitoring all aspects of laboratory operations including instrument and method performance and in tracking analyses. It is also important in confirming instrument performance at the time of specific analyses and in monitoring ongoing or periodic performance degradation and the steps taken to correct or prevent such occurrences. This document applies to all personnel involved in the preparation, control and use of laboratory notebooks and includes both physical logbooks and some aspects of electronic logbooks.

1.1.2 More specific instructions for maintaining logbooks can be found in pertinent SOPs, such as LTL-1007 "Maintaining Instrument Records and Logbooks" or LTL-1005 "Analytical Balances" or others specific to other laboratory operations.

1.2 Purpose

1.2.1 The purpose of this SOP is to define the practices used to maintain control and use of laboratory logbooks. This SOP is not intended as a specific description of any particular logbook type but covers the practices that must be in place for all logbooks employed at Laucks.

1.3 Definition of Terms

1.3.1 Logbook - Any bound, unbound or electronically presented document that forms a record of activities and pertinent data regarding an activity including but not limited to maintenance logs, standards logs, reagent chemical logs, analysis logs including instrument outputs (computer generated or strip chart recordings), balance and temperature logs, or any other regularly maintained record of activity.

## 2. Requirements

2.1 Control

2.1.1 Documents that specify quality requirements or activities affecting quality or evidentiary activities shall be controlled to ensure that correct documents are being used and properly archived when completed. This insures that logbooks can be found at later dates rather than lost or prematurely disposed. Electronic logbooks can be continuous and are not subject to physical degradation after extended use as are physical logbooks so they may not need to be archived for long periods of time and if so can still be kept in a readily accessible location, thus avoiding accidental loss or disposal.

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2.2 Maintenance

2.2.1 Quality assurance records (logbooks) shall be compiled and maintained in accordance with approved procedures.

2.2.2 Physical logbooks may degrade after extended use. Should they do so, the analyst must not allow them to deteriorate to the point that pages might be lost. The logbook should either be repaired or archived and a new logbook started. If this is done before the logbook is complete, the last page should be marked as such, dated, and initialed by the analyst.

2.3 Monitoring

2.3.1 Most logbooks should be periodically monitored to ensure they are being properly maintained and information is being correctly recorded.

2.3.2 Standard logbooks are electronic and must be filled in properly and signed-off before they are available on the iLIMS. Since all calculations are performed electronically, there should be no occasion where error could occur that will affect sample results. Therefore, the only monitoring is the check-off that must occur before the standard becomes available.

2.3.3 Run logs should be monitored at least semiannually by group supervisors or their designees. Run logs are printed out from instrument runs or are, in some cases, manually created but in most cases, there are no restrictions on their format or content so it is important that they be periodically inspected. Pages from these logbooks, however, should be at least checked for accuracy and completeness as the individual data packages are reviewed.

2.3.4 Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring.

2.3.5 At any time, whether during routine data review or specific logbook monitoring, if discrepancies are found, they should be corrected not only on the copies of logbook pages which may accompany a data package but on the original logbook page(s). If more systematic problems are found, the originator of the discrepancies must be re-trained in the proper documentation technique.

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## 3. Responsibilities

3.1 Quality Assurance Manager

- Maintains the logs for control of laboratory notebooks and provide control numbers and labels as required.
- Archives old physical logbooks in such a way as to be able to find them should the need arise, including being able to track when they are disposed (after at least 5 years from last entry).
- Reviews certain logbooks as part of the internal audit process (although only makes notations as part of the audit report and not part of the logbook record).
- Monitors satisfactory implementation of the requirements of this SOP

3.2 Responsible Supervisor

- Determine the format and content of notebooks used in their respective areas.
- In coordination with QA and the respective analyst(s) ensure that all laboratory notebooks are properly labeled, including the appropriate control number.
- Ensure that personnel are adequately trained in the proper use of laboratory notebooks
- Periodically review laboratory notebooks to verify satisfactory implementation of the requirements of this SOP. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself. Section 2 on requirements and Appendix II on typical review items provide more detail on this process.

3.3 Analyst

- Ensure that they are using the appropriate logbook and understand how to properly fill in the required fields. In general, this not only means entering correct information but that no pertinent information is left out or not entered in the assigned fields.
- Ensure that any new logbook has been given a logbook number by QA before beginning to use it.
- Ensure that the logbook is clearly identified with an instrument ID and purpose or other appropriate title which will enable the analyst to easily identify the logbook.

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- Ensure that if pre-printed logbook pages need to be modified, the modifications are approved by their supervisor.

## 4. Procedure

4.1 Notebook Structure

4.1.1 Laboratory notebooks may electronic, bound or unbound as described below. Most physical logbooks should be bound in some fashion but it is recognized that this is not always possible, such as for vendor service records. These records may be stored in 3-ring binders or other suitable notebooks but they may **NOT** be stored loose-leaf or in a simple manila folder.

4.1.2 In some instances, logbooks may be created from instrument printouts or other pages that do not lend themselves to being pre-bound. In these instances, the log sheets may be stored in a 3-ring binder or other storage until enough sheets have been accumulated to have them bound with the laboratory comb binder. Again, however they should **NOT** be stored loose-leaf or in a simple manila folder.

4.1.3 Where possible, especially if the logbook is unbound, if the output is specific to an instrument, that instrument should be identified on each page.

4.1.4 All physical logbooks whether bound or unbound must be controlled by QA as designated by the appropriate QA Book Number label (see example in Appendix 1).

4.1.5 Bound notebooks shall conform to the following:

- Where feasible, binding will be of a type that will make the removal and reinsertion of pages readily noticeable.
- If pre-printed and bound, all pages will be sequentially pre-numbered. If the format of the notebook permits the use of the reverse side of the pages (preferred), both sides of each page will contain a sequential page number.
- Each page of the pre-printed bound notebook will contain, as a minimum, the laboratory name, logbook title, and sequential page number. Other elements may also be necessary for any specific logbook.

4.1.6 Unbound notebooks shall conform to the following:

- Unbound pages will be contained in a binder or folder that prevents loss and provides protection from damage.

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- Each unbound page will contain a unique identifier (e.g., run number/date). For identification purposes, a continuous printout on fanfold computer paper requires only one identifier unless the sheets are separated.

- As noted above in 4.1.2, some unbound logbooks may eventually be bound if practical.

4.1.7 All notebooks will contain the following information on the cover:

- Laboratory name, Laucks Testing Laboratories, Inc.

- Control number assigned by the Quality Assurance Officer

- The department to which the logbook was issued

- The use of the logbook (i.e. balance calibration, instrument run-log, etc.)

- The department book number or title uniquely identifying that book, as required to identify the specific use of the book. This may include an instrument number or other logbook ID (such as a standards logbook ID). This is in addition to the QA logbook number.

- Since the labels used for logbook tracking are relatively small, it is often useful to write the title in large print on the cover to facilitate identifying the logbook from a distance.

- Start Date, the date on which the first entry was made

- End date, the date on which the last entry was made

4.2 Control of Logbooks

4.2.1 The QA Officer will maintain a master log of physical laboratory notebooks that contains as a minimum, the following information:

- Unique control number for each logbook

- Logbook title, which should reflect the type of information to be entered.

- Department to whom issued, for accountability only. A logbook will generally be assigned to a work station or function, and in no way is a laboratory notebook to be considered a "personal" notebook.

- Date issued, for accountability only.

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- Date closed, for accountability.
- Box in which the logbook was archived (if it has been archived)
- Whether or not that book/box has been disposed (at least 5 years after the last date of any book in the box)

4.3 Use of Laboratory Logbooks

4.3.1 The notebook is the basic document for recording information. Entries should be made into the notebook in real time, not written on scratch paper and transferred later.

4.3.2 Handwritten entries should be legible and entered in black or blue indelible ink.

4.3.3 Computer-generated data should be printed out and collected at appropriate times to represent the activities being recorded.

- Computer printouts may be either placed in unbound notebooks as described above, or inserted into bound notebooks.
- Computer printouts or other material inserted into bound notebooks must be securely fastened (tape is preferred) in such a way that removal and insertion of material can be determined readily.

4.3.4 When information from related activities is recorded in more than one notebook, provide adequate cross-reference information in all affected notebooks so that all pertinent data can be readily accessed.

4.3.5 Do not skip pages when entering data. For example, if data is not readily available for entry, do not leave space for later entry. Enter the data when it becomes available and provide adequate cross-references if required.

4.3.6 In cases where partial or complete pages must be left blank and not used, indicate the unused portion by placing a horizontal line at the beginning and end of the unused portion and connecting opposite ends of the horizontal lines with a diagonal, resulting in a Z-shaped figure. The individual striking out the blank area will initial and date the diagonal.

4.3.7 Errors or other changes must be deleted in a similar fashion or with a single-line cross-out which has been initialed and dated. **No erasures, overwriting, white-out or multiple-line cross-outs (blacking out) are acceptable.**

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4.3.8 When pre-printed formats are used and all possible entries are not required, the remaining blanks may be struck out with a Z as described above, or entries such as N/A may be placed in the unused blanks.

4.3.9 The individual entering information into the notebook shall initial and date each page used, or in the case of logbooks with ongoing records which do not occupy the entire page, such as maintenance logs or balance logs, each individual entry.

4.4 Supervisory Monitoring of Laboratory Logbooks

4.4.1 Standard logbooks and run logs should be monitored as discussed in section 2.3 by group supervisors or their designees. Maintenance and other logbooks need only be reviewed annually, unless previous review has demonstrated inadequacies in the logbook which require more frequent monitoring. This activity may be assigned to another individual but should not be the same individual who regularly completes the log itself.

4.4.2 Logbooks should be reviewed using the review items provided in Appendix II, although it is not necessary to actually document the review using this checklist.

4.4.3 Errors should be formally brought to the attention of the responsible individual through the use of Corrective Action Forms. If errors are correctable or items can be corrected for legibility problems, they should be corrected using the proper error correction technique.

4.4.4 Logbooks that have been reviewed are marked with a fluorescent yellow or other colorful label that looks similar to the label in Appendix III.

4.5 QA Monitoring of Laboratory Logbooks

4.5.1 The QA Officer will verify during periodic audit and surveillance activities that notebooks are properly completed and maintained. This will generally be done approximately annually as part of routine audits. This observation does not preclude the requirement for supervisory review.

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## Appendix I

### Example QA Logbook Label

**Laucks**
Testing Laboratories, Inc.

QA Book No.: _____

Issued To: _____

Used For: _____

Dept. Book No.: _____

Start Date: _____

End Date: _____

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## Appendix II

### Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced before it completely falls apart.

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## Appendix III

### Example Logbook Review Label

Logbook pages _____ through _____
have been reviewed for completeness and
spot-checked for accuracy.

Initials: Date:

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1020

Title: **The Integration of IC, GC, HPLC, and GC/MS Peaks**

Revision history:

| Number | Date |
|---|---|
| 0 | 08/26/96 |
| 1 | 08/21/97 |
| 2 | 05/02/02 |
| 3 | 04/30/07 |

Revised by: _____ Date: 5/7/07
Monica Carr, Technical Reviewer

Reviewed by: _____ Date: 5/8/07
Harry Romberg, QA Officer

Approved by: _____ Date: 5/8/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the integration process for chromatographic data, the procedures for manual integration, and the procedures for documenting manual integration.

1.1.2 Integration identifies peaks found in the data collected during data acquisition and characterizes them. The software uses the integrated peaks to determine the identity and quantity of compounds in the samples. The peak area, peak height, peak type, baseline, and retention time of each peak in a chromatogram are determined by integration. It is expected that situations will arise when the automated quantitation provide inappropriate quantitations. This normally occurs when there is compound co-elution, baseline noise, or matrix interference. Some peaks, due to limitations of the software, will need to be manually integrated. The manual integration process must be documented as outlined in section 5 of this SOP.

1.1.3 Manual integrations have been a source of serious concerns throughout the laboratory industry as a tool of fraud. There have been labs as well as individuals who have been penalized due to improper manual integration, or inadequate documentation that their actions were appropriate. Therefore, it is imperative that proper and honest and completely documented procedures are followed.

1.1.4 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Definition of Terms

1.2.1 Manual Integration - any intervention by an analyst or supervisor to change the peak area, peak height, baseline, peak type, or retention time of a chromatographic peak.

## 2. Software

2.1.1 GC Acquisition-HP/EZChrom Version 6.6
2.1.2 GC/MS-Teknivent/EnviroQuant/Chemstation Versions B.01.00, D.00.01 and D.01.00 Sp1
2.1.3 GC-Target Version 4.02

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## 3. Responsibilities

3.1 Analyst

3.1.1 The analyst is responsible for reading and understanding this SOP and that which is applicable to the method of analysis. The analyst must also perform and document all manual integrations as specified in this SOP.

3.2 Supervisor

3.2.1 The supervisor or designated data reviewer must verify that all manual integrations are performed according to this SOP. The supervisor or designated data reviewer must document that this verification has occurred per the applicable Data Review, Validation, and Reporting SOP.

## 4. Operation Procedures

4.1 Data System Integration

4.1.1 The ideal chromatogram has perfectly symmetric peaks separated from each other with periodic baseline points. It is common to encounter split peaks, deformed peaks, merged peaks, sloping baselines, noise, spikes, shoulders on peaks, and a host of other calamities.

4.1.2 To maximize the chances of obtaining ideal chromatograms, first optimize the chromatographic conditions.

4.1.3 Peak recognition and integration sequence - As the data system scans the data, it examines the slope (vertical distance between points) and curvature (positive or negative). So long as these remain within preset bounds data is interpreted as the baseline. If the bounds are exceeded, a peak may be starting. If the condition persists, the integrator decides that it is on the upslope of a peak.

4.1.4 The curvature changes to negative about halfway up the peak. This is the inflection point where the peak starts to round over approaching the apex. Passing the top, the slope becomes negative and the integration is on the downslope. Another inflection point comes on the downslope and finally the peak returns to the baseline (Figure 1).

4.1.5 Finite width of integration slices - The slope changes from positive to negative at the top of the peak. However for area slices having finite width the data system can only determine which slice contains the peak apex. To get better values for the retention time and peak height,

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integrator takes the slice containing the-apex and one slice on either side, fits them to a quadratic equation, and solves the equation to find the highest point (Figure 2).

4.1.6 Optimizing peak recognition - The best conditions for recognizing isolated symmetric peaks on a quiet baseline is to match the peak width parameter to the measured width of the peaks at half height. Threshold should be a few units less than the highest value still capable of detecting the peak. When peaks cluster together or the baseline slopes or is noisy, these ideal values must be modified. Figure 3 shows the effects of changing the values.

4.1.7 Manual integration - It is important that the analyst is familiar with the compounds that are routinely analyzed. Knowing the response and peak shape of the standard is important for consistency in integration. It is best to optimize the method to process data so that manual intervention is minimized and peak integration is more consistent. If manual integration is performed, it is important to be consistent for a given analyte in the standards, blanks, spikes, and samples.

4.1.8 Allowable manual integrations - Some common reasons for manual integration are:

4.1.8.1 Split peaks (attachment 1)

4.1.8.2 Tailing (attachment 2)

4.1.8.3 Retention time shifts (attachment 3)

4.1.8.4 Mis-identification (attachment 3)

4.1.8.5 Merged peaks (attachment 3)

4.1.8.6 Secondary ions or qualifier ions (attachment 4)

4.1.8.7 Baseline shifts (attachment 5)

4.1.8.8 Skimming versus dropped baseline (attachment 6)

4.1.9 Improper manual integrations - These practices are not allowed and warnings up to and including termination of employment will follow any documented cases of improper manual integration. If you are unsure about a manual integration ask your supervisor or QA.

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4.1.9.1 Adding area by including other peaks. (attachment 7)

4.1.9.2 Improper baseline - this includes the practice of having the baseline moved up the side of a peak to decrease the area. (attachment 8)

4.1.9.3 Changing a proper integration to make the peak "in".

4.1.10 Special rules for fuel analyses (*e.g.*, gasoline, diesel) - The integration of these multi-component analytes requires special integration rules. The area of all peaks (with the exception of the surrogates) and the area of the non-resolved components (hump) are grouped together to determine the quantity of the analyte. The baseline is fixed at the start and held at a constant level for the entire run. When integration of the baseline for fuel analyses is performed, the quantitation report will not be dated and initialed by the analyst. Instead an explanation of the baseline integration will be documented on the quantitation report or in the appropriate SOP and discussed in the sample narrative. Some acceptable multi-analyte integrations are shown in attachment 9.

## 5. Documentation

5.1.1 When any manual integration is performed, a graphic copy of the peak with the integration marks is generated and put into the folder with the chromatograms and quantitation reports for that sample. The three different software systems used in the laboratory are listed below with the commands for generating the copy. On the quantitation report the analyst must initial, date, and give a brief description of the reason for the manual integration (table 1). In addition, the analyst must provide chromatograms from both before and after the manual integration. The manual integration must also be documented in the associated sample narrative. The supervisor or his/her designee will look at each manual integration during data review and complete the summary on the data review or QC checklist.

5.1.2 Target: After changing the integration, exit and save in Target Review.

5.1.3 GC/MS Teknivent EnviroQuant/Chemstation: After using Qedit to change the peak click on 'Graphics Report to Printer'

5.1.4 EZChrom: After changing the integration, toggle the “Reanalyze” key and print.

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Table 1

Manual Integration Key

M = Manual integration due to missed peak or irregular peak shape.
MS = Manual integration due to split peak.
MR = Manual integration due to retention time shift.
MI = Manual integration of correct isomer.
MT = Manual integration due to peak tailing.
MB = Manual integration due to irregular baseline.

## 6. REFERENCES

6.1.1 Target Manual, Thru-Put Systems, Inc., Target Compound Analysis Software User's Guide, 1994, Version 4.02.

6.1.2 Envirolink User's Manual, TEKNIVENT, 1994

6.1.3 HP Chemstation, Version B.01.00, D.00.01, D.01.00

6.1.4 EZChrom Chromatography Data System, Scientific Software, Rev. 6.6, 1995.

6.1.5 Wisconsin DNR Newsletter, October 1994.

6.1.6 SOP# LTL-1018, Standard Operating Procedure for Review and Approval Practices for Validatable Packages.

6.1.7 SOP # LTL-8005, Standard Operating Procedure for GC Gas/BTEX Data Review.

6.1.8 SOP #LTL-8004, Standard Operating Procedure for GC Volatiles Data Review.

6.1.9 SOP #LTL-8001, Standard Operating Procedure for GC Hydrocarbons Data Review.

6.1.10 SOP #LTL-8302, Standard Operating Procedure for HPLC Ordnance 8330 Data Review.

6.1.11 SOP #LTL-8201, Standard Operating Procedure for GC/MS VOA Data Review.

6.1.12 SOP #LTL-8202, Standard Operating Procedure for GC/MS Semivolatile Data Review.

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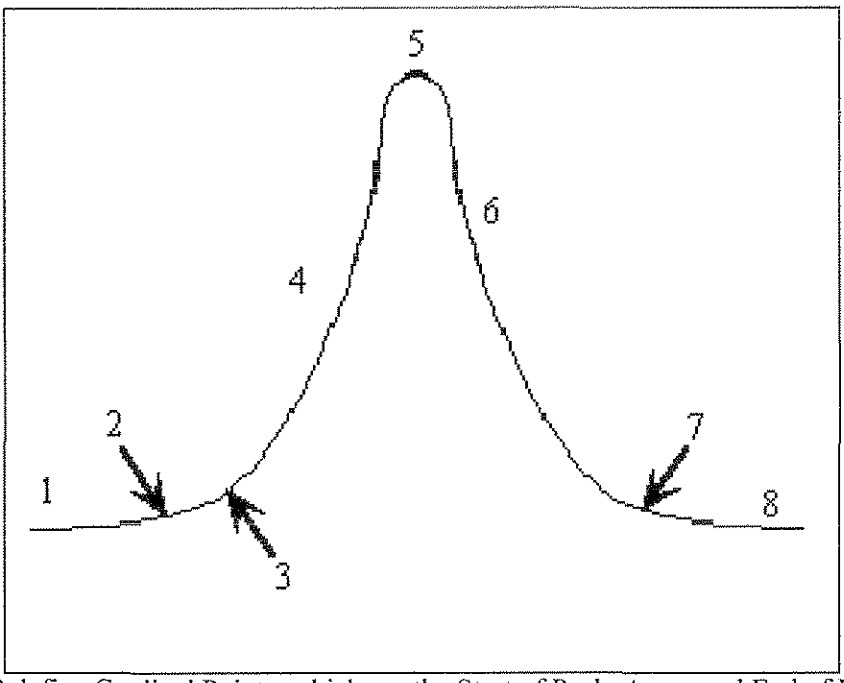
## Appendix I

### Fig 1.

The sequence for finding a positive peak is:

| | |
|---|---|
| 1. Slope and curvature within limits | track baseline |
| 2. Slope and curvature above limits | perhaps a peak? |
| 3. Slope remains above limit | here's a peak! |
| 4. Curvature becomes negative | front inflection point |
| 5. Slope becomes negative | top of peak |
| 6. Curvature becomes positive | rear inflection point |
| 7. Slope and curvature within limits | approaching end of peak |
| 8. Slope and curvature remain within limits | end peak, track baseline |




Steps 3,5, and 8 define Cardinal Points, which are the Start of Peak, Apex, and End of Peak Respectively

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**Fig 2.**

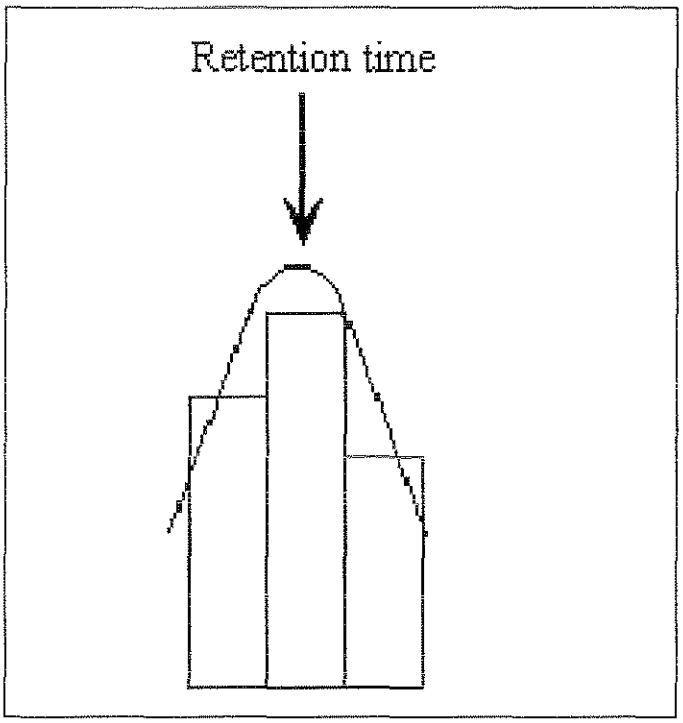

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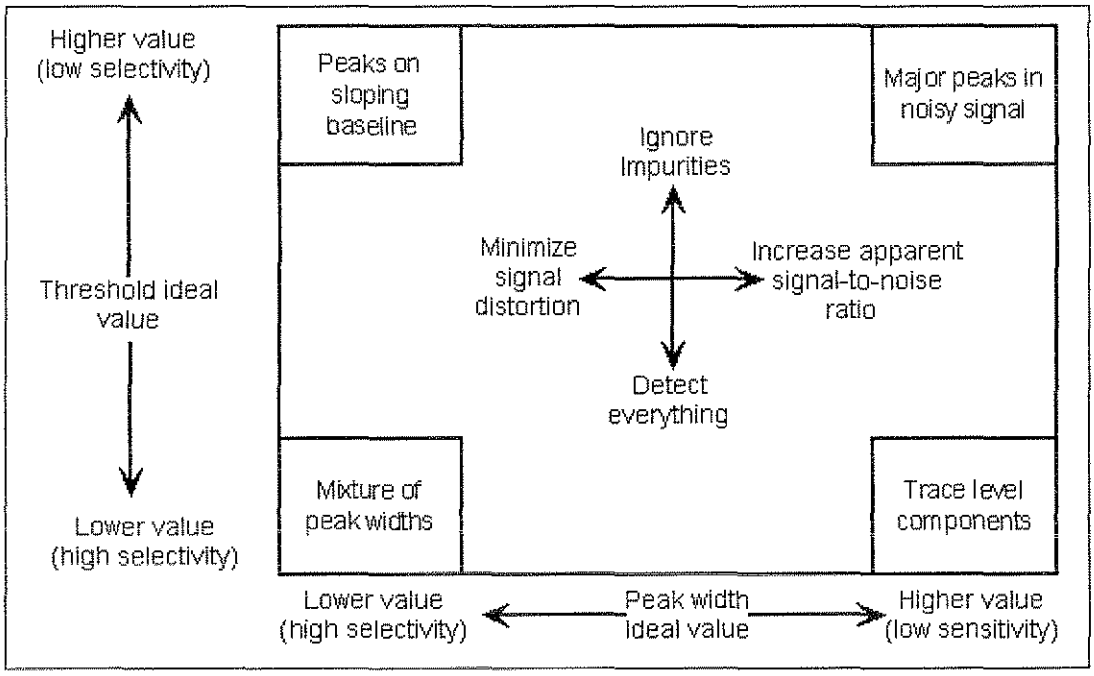
**Fig 3.**



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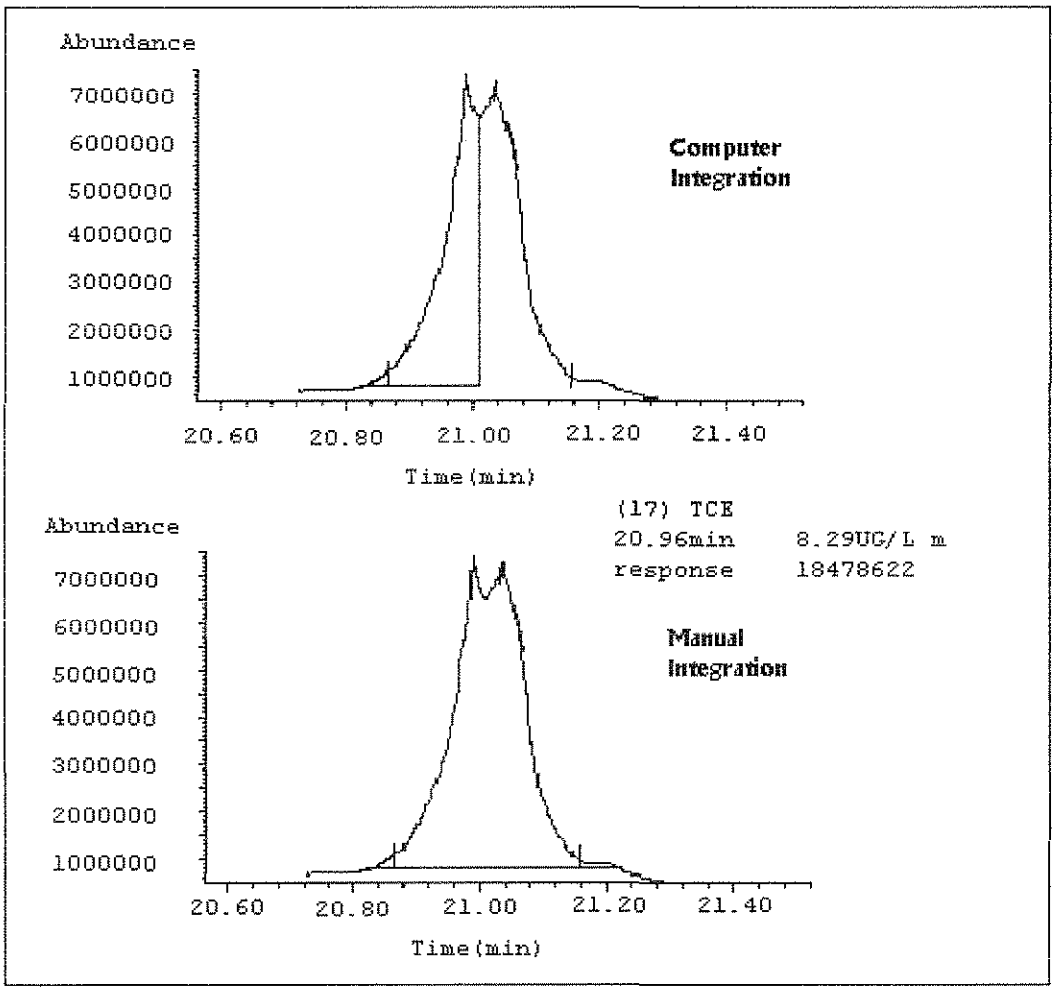
## Appendix II

### Attachment 1




Allowable Integration

Split Peaks

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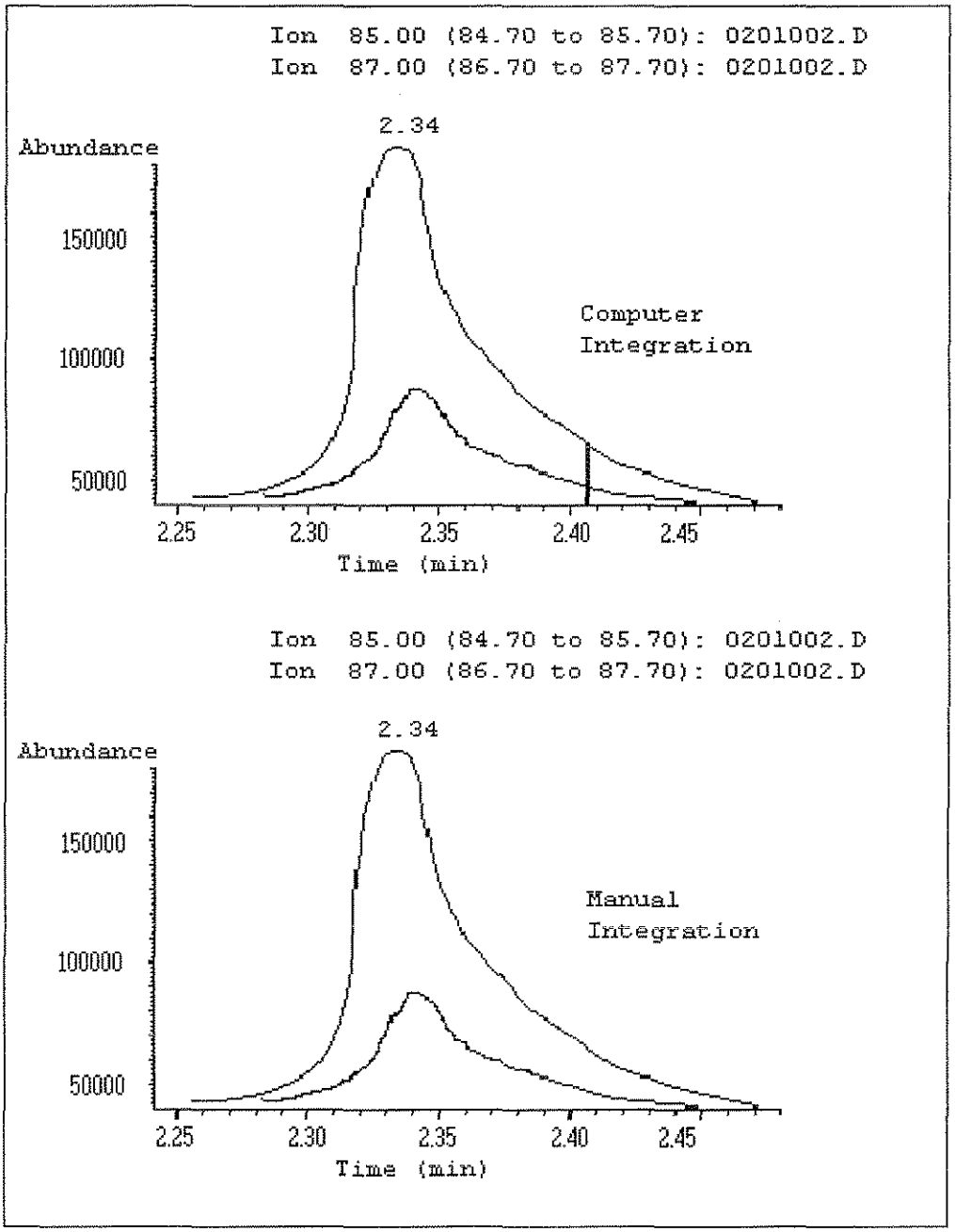
**Attachment 2**




Allowable Integration

Tailing

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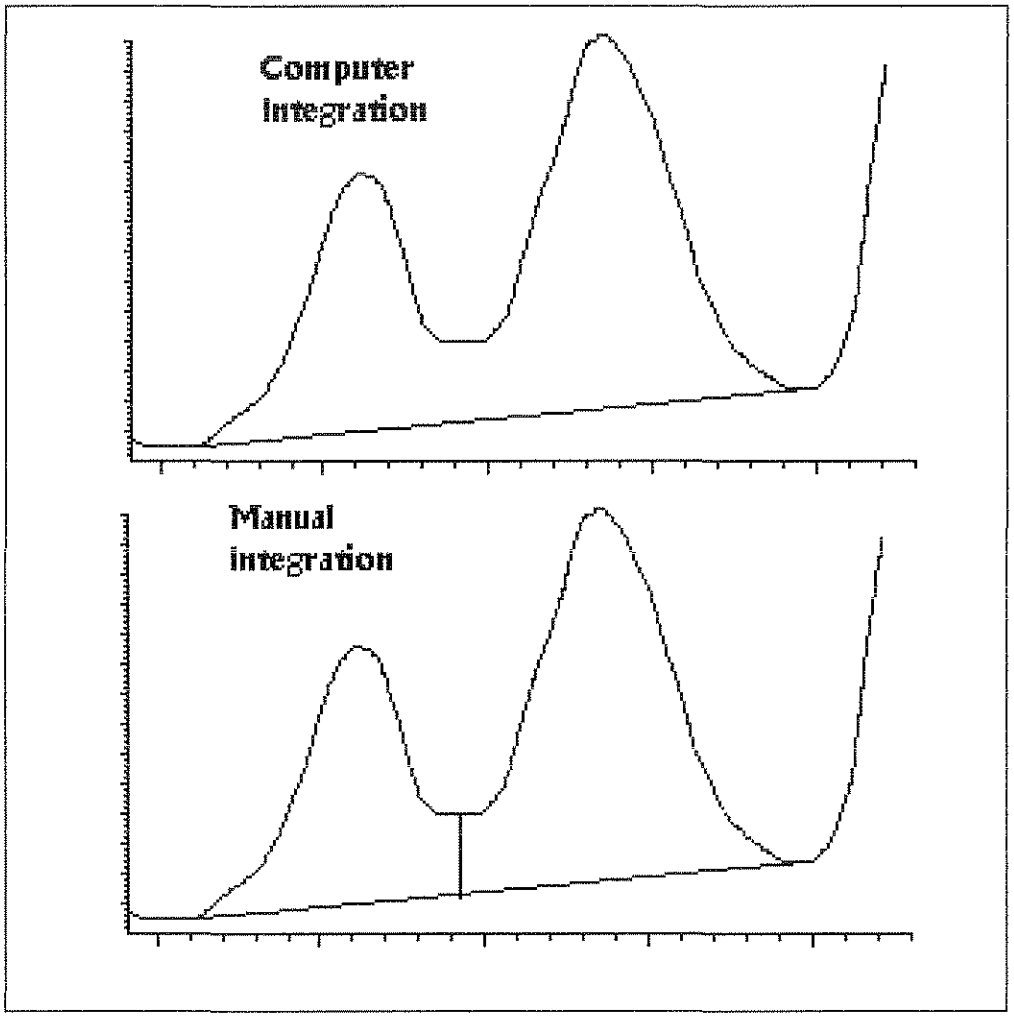
**Attachment 3**




Allowable Integration

Retention Time Shift - Mis-Identification - Merged

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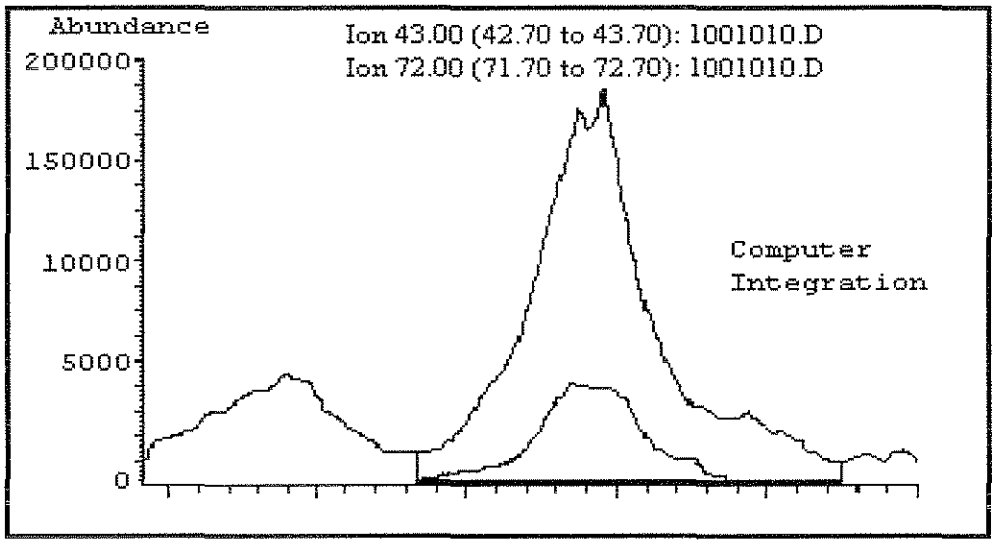
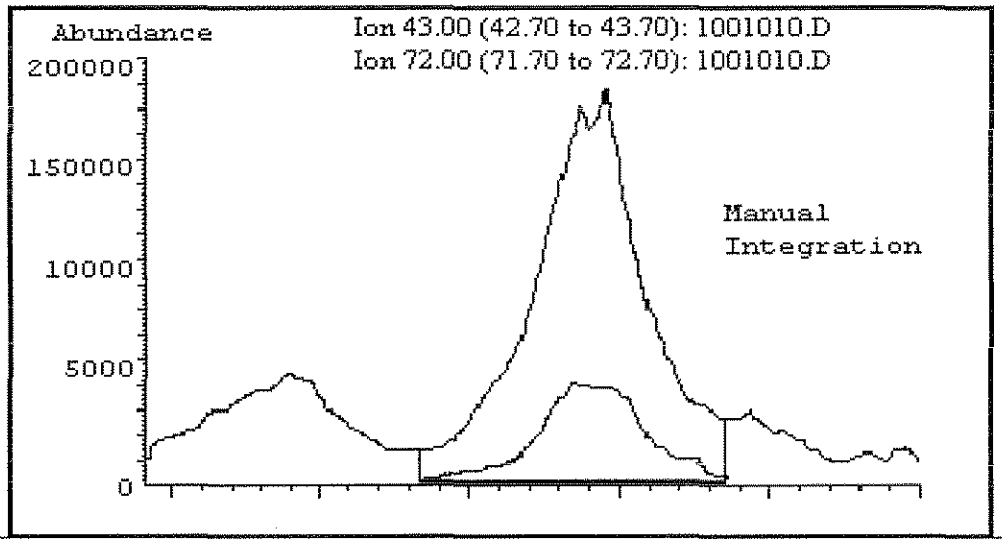
**Attachment 4**




Allowable Integration

Secondary or Qualifier Ions

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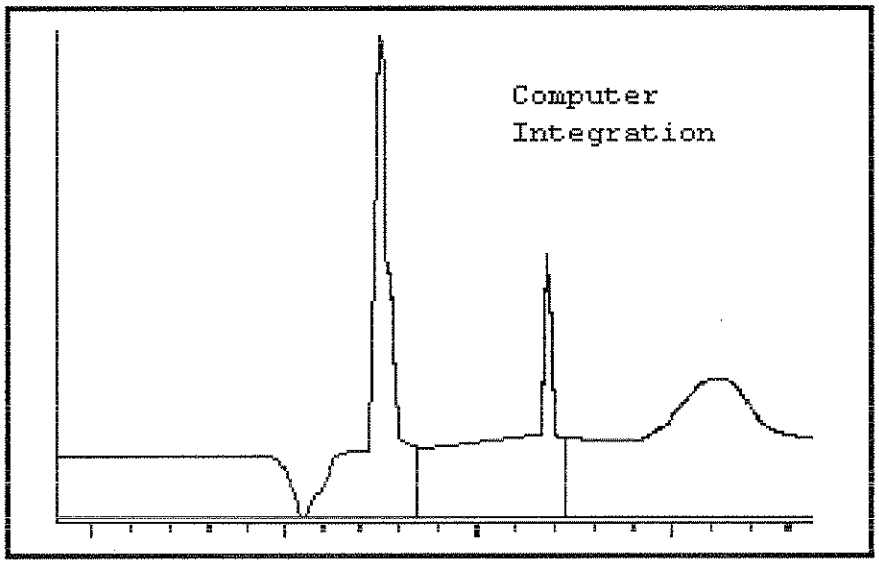
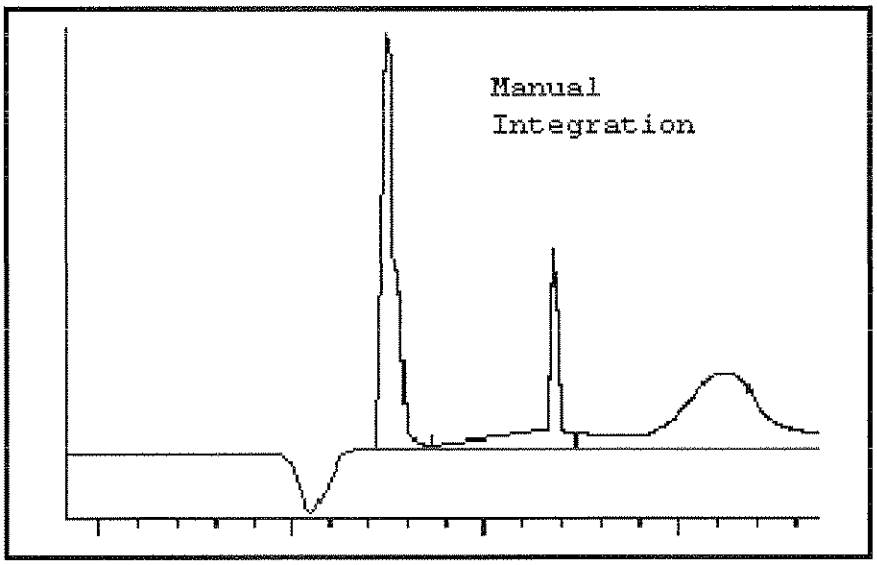
**Attachment 5**




Allowable Integration

Baseline Shift

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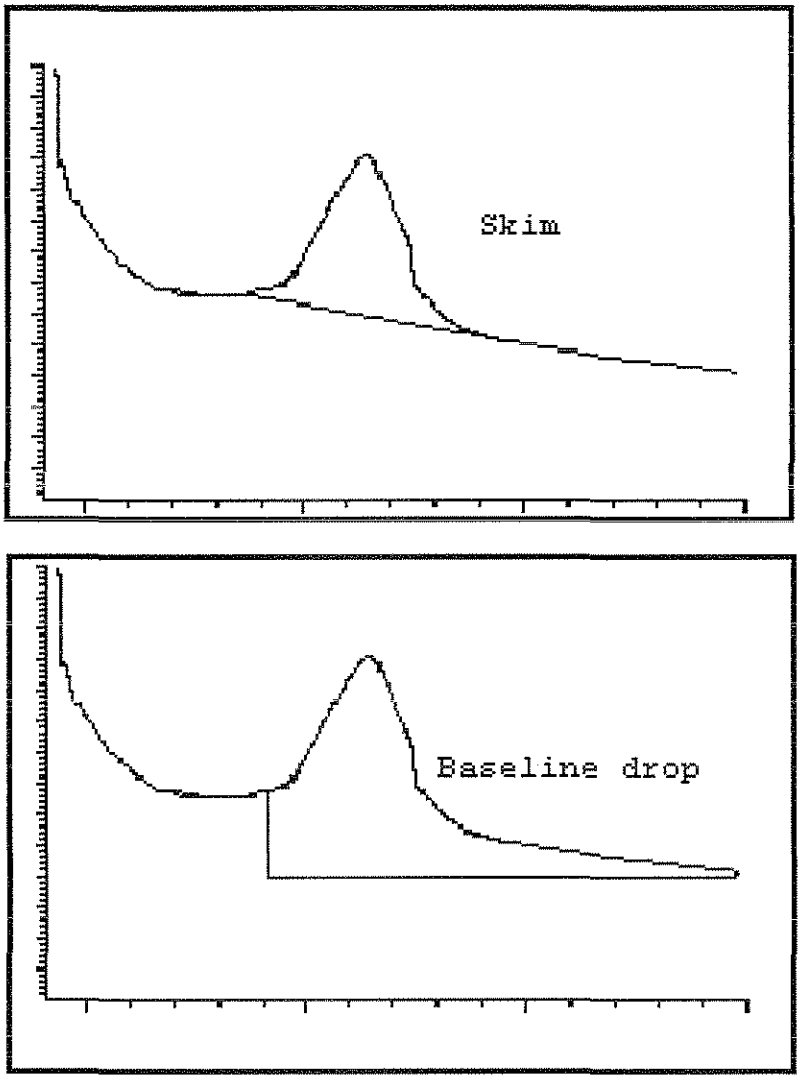
**Attachment 6**




Allowable Integration

Skimming (manual) vs. Baseline Drop (computer)

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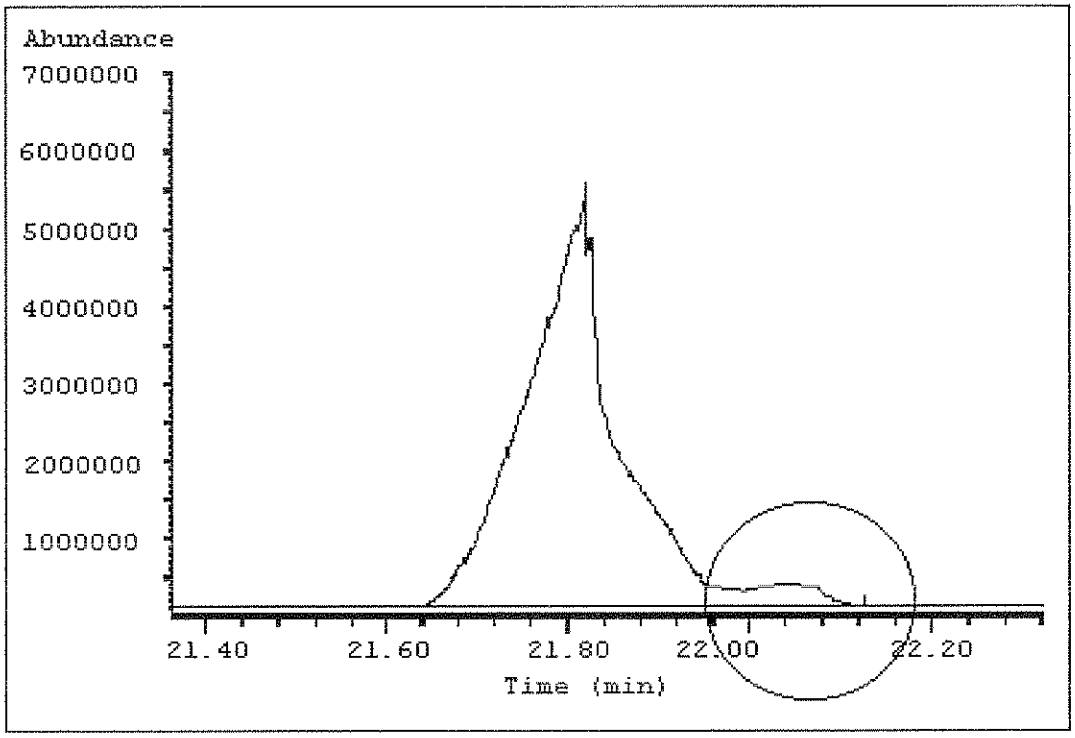
**Attachment 7**




**Improper** Integration

Adding Area by Including Other Peaks

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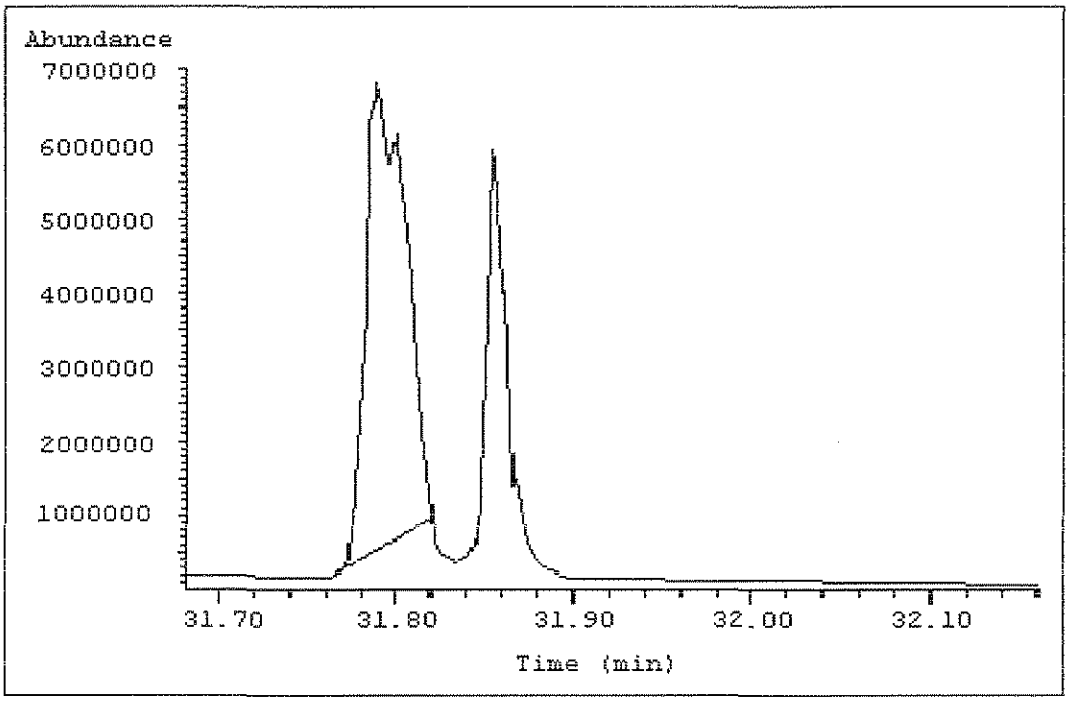
**Attachment 8**




**Improper** Integration

Baseline Moved To Decrease Peak Area

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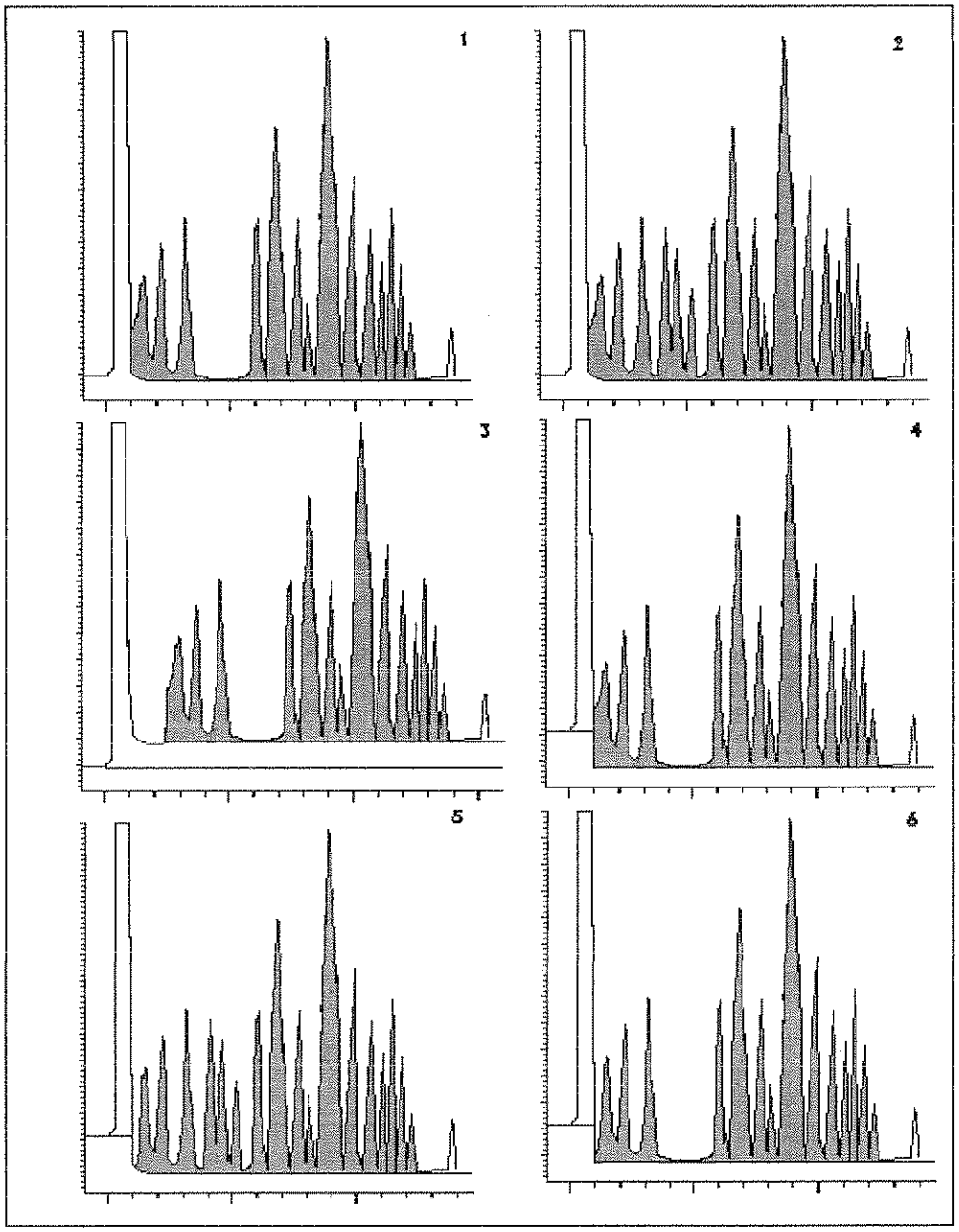
**Attachment 9**




Acceptable Methods of Integration
Grouped Peaks

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

| Number | Date |
|---|---|
| 1 | 4/10/91 |
| 2 | 9/11/91 |
| 3 | 4/26/94 |
| 4 | 4/09/97 |
| 5 | 5/12/98 |
| 6 | 10/10/01 |
| 7 | 10/18/04 |
| 8 | 12/15/05 |
| 9 | 5/21/07 |

Written by: Harry Romberg Date: 5/21/07
Harry Romberg, Quality Assurance Officer

Approved by: Hugh Prentice Date: 5/22/07
Hugh Prentice, Laboratory Director

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to describe the laboratory waste disposal scheme currently in place at Laucks. The primary waste streams described include solvents, PCB oil wastes, COD waste, soil samples, aqueous acidic waste, and fluorescent light bulbs. This SOP only covers handling of the waste from the point of collection.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the techniques described. As part of their training for analytical tasks which generate related wastes, each analyst must be trained to properly dispose of the waste or to consolidate it at the appropriate collection point.

1.1.3 This SOP generally does not cover handling of the waste up to the point of disposal.

1.1.4 This SOP does not address the specifics of the occasional disposition of labpacks. As these cases are not common, the hazardous waste vendor is contacted each time a labpack disposal is required in order to insure the latest requirements are met. Most aspects of labpack disposal mirror other operations outlined in one or more areas of this SOP.

1.1.5 Appendix III, entitled *"A Quick Primer on Hazardous Waste Management"* contains a brief description of what is expected of individuals in the laboratory in terms of hazardous waste disposal. This is primarily to be used as a reminder and may be handed out independently of the entire SOP. Staff members who handle hazardous waste should also read this SOP.

## 2. Equipment List

2.1 Equipment

2.1.1 The equipment necessary to properly dispose of laboratory wastes varies with the type of waste. In general, an appropriate container, packing material, and safety equipment (including clothing, eye wear, and respirators) is required.

## 3. Safety precautions

3.1 Safety Precautions

3.1.1 Solvent wastes may contain materials flammable at room temperature or lower. Caution should be taken to avoid flames and sparks when in the presence of or handling these wastes.

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3.1.2 COD and other acidic or alkaline wastes may contain materials which will burn the skin, eyes, and/or mucous membranes if improperly handled. Precautions should be taken to avoid accidental contact.

3.1.3 All wastes may contain materials which can have both known and unknown long-term health effects. COD wastes, for instance, contain high levels of mercury and chromium salts. Direct contact should be avoided through the use of proper clothing and eye wear, even if no immediate danger is obvious. In the case of volatile solvents and other materials, handling should be done in a well-ventilated area and the exposure to vapors minimized. Where strong fumes are unavoidable, a carbon-filter or other respirator should be worn.

3.1.4 All people who handle waste products or the original reagents should be aware that the laboratory provides safety equipment and has a file containing Material Safety Data Sheets (MSDSs) on all laboratory chemicals in support of OSHA and other safety programs.

3.1.5 In addition to having obvious short-term health effects as noted above and in the MSDSs, these waste products may have serious longer term health effects such as hormone disruption or carcinogenicity. They should be handled carefully in order to avoid such contact and minimize potential health risks.

## 4. Operation procedures

### 4.1 Operations Appropriate to All Collection Areas

4.1.1 Land Disposal Restriction Forms (LDRs), manifests and other paperwork are not extensively discussed in this SOP because the disposal vendor deals with this aspect of the paperwork. It will only be necessary for the person who will be asked by the vendor to sign these forms (usually QA) to check that the information on the forms is accurate and to sign the form.

4.1.2 All waste requiring a Hazardous Waste disposal sticker and manifest will be marked with one of two EPA Hazardous Waste Site numbers. All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608. It is only necessary that these numbers appear on the containers before they are shipped. It is not necessary on drums which have yet to be transported. The labels themselves, however, must be placed on the containers prior to use.

4.1.3 The Hazardous Waste Sticker must be labeled with the proper DOT shipping name, even though the disposal company will usually replace the label before shipping. The

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proper shipping names are listed below in the applicable sections of this SOP. This item **IS** necessary on all drums as soon as any waste at all is dumped into them.

4.1.4 All collection drums **must** be marked with an appropriately filled out Hazardous Waste sticker (see Appendix I). It is only necessary for Laucks staff to fill in the date that collection was **started** and the contents of the drum in the appropriate space. Hazardous Waste cannot be accumulated for longer than 90 days before it must be disposed in the 940 facility as that building is considered a large quantity generator. The 921 building qualifies as a medium quantity generator, however, and waste may be held for up to 180 days before disposal. Therefore, do **not** mark the date on the drum until collection is started so as to maximize the allowable time until disposal. This applies to the 55 gal. drums of solvent in the 921 building and the 30 gallon drum in the 940 solvent locker. All other containers are considered satellite collection points and need only be dated when the containers are **filled**. This sticker will be replaced by the transporter when they arrive to transport the waste to an approved disposal facility. The replacement sticker will contain all of the information required for transport and disposal.

4.1.5 All collection drums containing liquids should have secondary containment. That is, the primary collection drum should sit in a tub or bermed space so that if the container suffered partial or complete failure, there would be sufficient space in the secondary container to hold all of the liquid and not allow it to drain to the sewer.

4.1.6 In addition, corrosive and flammable waste collection drums must have a sticker which indicates their corrosive (8) or flammable nature (3) (see Appendix II). Soil drums are designated with a general hazard (9) label.

4.1.7 Once a material has been designated as waste and disposed into the designated drum, that drum must not be stored for longer than 90 days from the date marked on the drum as designated above if located in the 940 building (because 940 is a Large Quantity Generator, LQG) and 180 days if located in the 921 building (which is a medium quantity generator, MQG). This is rarely of concern at Laucks because transport is generally scheduled for most wastes within much less time than the required maximum storage time. These time requirements do not apply to satellite containers which are intermediate storage locations before disposal to the final designated storage area.

4.1.8 When collection drums are full or the time limit is approaching, the Quality Assurance (QA) Department must be notified. This department, at the time of this writing, is responsible for contacting the appropriate approved transporter and insuring proper disposal takes place. QA does weekly inspections of the laboratory with the express

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purpose of observing the hazardous waste areas and making safety related observations, but those filling waste containers should still keep QA informed.

4.1.9 All questions or concerns regarding hazardous waste operations should first be directed to QA who will determine the appropriate course of action.

4.1.10 QA or the designated waste manager will also be responsible for insuring that signed manifests are received from the disposal facility within 35-days of shipment. This is easily done by the organization of our hazardous waste files for both of our facilities.

4.2 Mixed Solvent Waste

4.2.1 This waste stream is primarily composed of methylene chloride with some acetone and hexane and potentially small quantities of other solvents or dissolved products. The collection point for all of this waste in the 921 facility (Extractions) are the 55 gallon drums in the solvent locker.

4.2.2 In the 940 facility, the waste is slightly different with significant though less methylene chloride but also containing more acetonitrile, some methanol and significant quantities of water. The designated collection site is a 30 gallon drum in the 940 solvent locker.

4.2.3 Small, 5 gallon or less, containers of other mixed solvent waste may be collected as satellite accumulation units in the 940 inorganics or organics instrument preparation areas or near the HPLC but these **must** be transported to the primary drums when full. Likewise, waste solvent bottles are kept in some hoods in the 921 Extractions facility. Satellite accumulation containers must be kept closed when not in use and must be marked with the words "Hazardous Waste" or with other words that identify the contents of the container. This will most conveniently be done by using a blank Hazardous Waste Sticker. Open solvent containers are not allowed except when actually in use.

4.2.4 All waste solvent containers whether in final or satellite accumulation areas, must be in secondary containment. This means that the primary container is sitting in another container such that if the primary containment fails catastrophically, the secondary containment will be able to hold the entire contents of the primary so that it doesn't flow down a drain or into the environment. The 55 gallon and 30 gallon solvent drums are stored in rooms with bermed doorways so this is not a problem. Satellite containers, however, will usually need separate containment.

4.2.5 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of **"Waste Flammable Liquids"**.

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4.2.6 Although methylene chloride is non-flammable, other components of these waste drums may be highly flammable. Thus, all of the waste solvent containers must be labeled with the flammable hazard (3) sticker (see Appendix II).

4.2.7 About 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.8 When 3 full 55 gallon drums of this waste have been accumulated at 921 (or two drums and a drum of soil), or 1 full 30-gallon drum at 940, or the requisite number of days have passed since the beginning of collection of the oldest accumulated drum, QA must be contacted to arrange for transport and disposal.

4.2.9 At the time of this writing, Laucks uses Veolia as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. Veolia is able to recycle most of the 921 waste stream for its methylene chloride content but the 940 site has too low a content of this solvent to be worth it so it is incinerated instead.

4.3 Chemical Oxygen Demand (COD) Waste

4.3.1 The primary constituents of this waste are sulfuric acid, water, mercury, silver, and chromium (both tri- and hexavalent). The collection point for this waste is in the inorganics area where CODs are analyzed. These analyses are conducted in small pre-packaged tubes. The reacted tubes are not considered to be waste until they are poured out of the tubes into a collection container.

4.3.2 Collection containers must be labeled with a Hazardous Waste sticker as previously noted. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.3.3 In addition to the hazardous waste sticker, these containers should be labeled with a corrosive (8) sticker (see Appendix II) as previously noted.

4.3.4 This waste is only accumulated at the 940 building and thus may not be held for more than 90 days from filling of the satellite 5-gallon collection container. Immediately upon filling the container it must be tightly closed, dated, and transported to the acid storage locker. QA must be immediately contacted to arrange for transport and disposal within the allowable timeframe. While being filled, the 5-gallon collection container must be in secondary containment which consists of a washtub in which the collection container sits. **Disposing of any solutions containing anything other than the primary components of COD analysis noted in 4.3.1 must first be cleared with QA in order to avoid a hazardous reaction.**

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4.3.5 At least 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.3.6 At the time of this writing, Laucks uses Veolia as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis.

4.4 Soil Sample Disposal

4.4.1 Soil waste drums must be labeled with a hazardous waste label and identified as **"soil contaminated with lead"**. It must also have a general hazard (9) sticker placed right next to the hazardous waste label on the upper half of the drum.

4.4.2 State law allows a laboratory to store samples indefinitely, until they are considered waste and disposed. The laboratory is also allowed to store a 55 gallon drum for at least 90-days (depending on our generator status…generally 180 days for the 921 site) after it has been **filled and dated**. The drum must be tightly sealed and an accumulation date written on the hazardous waste label immediately after it has been filled.

4.4.3 Each drum used for soil waste disposal must be clearly marked with an identifying number which will be used to track which drum contained which samples. This will generally be done with a bar code generated from the LIMS. When samples are scanned-out from their storage areas for disposal, they should be scanned or assigned to the marked drum. This will enable the laboratory to track which samples were disposed in which drum.

4.4.3.1 Laucks frequently receives soil samples from outside the continental U.S. This requires a USDA APHIS soil import permit. Samples from specific area within the mainland U.S. may also qualify (see map provided to PMs and sample control). The permit requires that we heat-treat many of these soils in order to kill foreign nematodes or other potentially harmful organisms that may be in the soil. All non-domestic soils are transported to an oven designated for this purpose in the 940 building warehouse where they are heated to over 250°F for at least 2 hours. The samples are scanned or assigned out of their storage area into the oven (which has been given a bar-code / number), heat treated, and then assigned to the proper disposal drum.

4.4.3.2 On occasion, Laucks also receives samples with high PCB content. These are separated into 2 different drums. Those with <2 mg/kg total PCBs are treated just as any other soil (including heat treatment if the sample is from outside the U.S.). **Note:** High PCB samples should NOT be heat treated in the laboratory oven so that they will not contaminate the oven or carry over into the lab. Those with

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>2mg/kg total PCBs are actually considered to be in one of 3 classes, those >2mg/kg but <50mg/kg, those >50mg/kg but <500mg/kg, and those >500mg/kg. It is rare that any soil is in either of the highest two categories and Laucks incinerates all of these samples in order to destroy the PCBs. So, at this point, for treatment purposes, Laucks does not really distinguish treatment by the level of PCBs present. Incineration also serves to meet and exceed the heat-treatment requirement of the USDA permit. Incineration is a much more costly method of disposal than stabilization, which is how most of the other soils are treated, so care should be taken to properly separate those soils requiring this special treatment from those requiring the "normal" treatment.

4.4.3.3 Soil sample bottles will should have their lids removed. If they are clean, they may be disposed in the regular garbage. If not, they should be disposed in the same way as the contents of the bottle (heat treated if necessary and placed in the disposal drum). If the contents of the jar are easily poured into the drum, this is done and the jar disposed in the waste glass. If not, jar and all will then be disposed in the waste drum.

4.4.3.4 When the drums are disposed, it will be necessary for the laboratory representative who signs the manifest to mark the drum identity on the manifest, although this only needs to be on the laboratory copy if the transporter does not want this information to appear on their copy of the record.

4.4.4 QA must be notified when the drum is full in order to arrange for disposal in a timely manner. This timeframe is not of major concern because there are always Hazardous Waste pickups scheduled within any 90 day time period.

4.4.5 At the time of this writing, Laucks uses Veolia as the vendor of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis. The vendors solidify (stabilize) these soils prior to landfilling in an approved landfill.

4.5 Acidic Waste

4.5.1 All acidic waste must be neutralized before disposal. This includes acid-preserved samples, digests, acid-washings or soaking bathes, etc. unless they contain high levels of heavy metals (which can be disposed with our COD waste if they are not too voluminous, <500 mLs.).

4.5.2 The wastes are poured into a stoppered sink and neutralized with sodium bicarbonate. The resulting neutralized liquid is flushed down the domestic sewer with large quantities of tap water.

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4.5.3 Their neutralization and disposal must be documented as it impacts Laucks hazardous waste generator status. This is done in a designated logbook kept near the neutralization sink where the date and final volume disposed are recorded. Note, this volume should only include the volume that was actually neutralized and not that of the water used to flush the neutralized solution down the drain.

4.6 Fluorescent Lights

4.6.1 Fluorescent light bulbs are also disposed as hazardous materials. They are in a category termed "universal waste" and are collected in a well marked box similar to that in which they were purchased. When the box is full, it is sent to our hazardous waste vendor or to another recycling firm. This waste stream does not need to be disposed within any particular time frame but should be properly taken care of as soon as the box of bad bulbs is full. Care must be taken to not break the bulbs as they are coated on the inside with hazardous fluorescent material often containing high levels of mercury.

4.7 PCB Oil Waste Disposal

4.7.1 Laucks no longer analyzes many oil samples for PCBs. Thus, this is a very small and infrequent waste stream. However, discussion is presented here in order that there be some documented course of action when it is necessary to dispose of these materials.

4.7.2 All oil samples which are analyzed for PCBs or otherwise known to contain PCBs are treated as PCB oils. No effort is made to distinguish those that actually do contain PCBs.

4.7.3 These oils are accumulated in a 5 gal. drum located in the Extractions laboratory warehouse. This metal drum is stored inside of the lower half of a cut-off plastic 55 gal. drum which fulfills the federal requirements for secondary containment during storage.

4.7.4 When a full drum has been accumulated, Eastern Electric is contacted for pickup and disposal. A signed receipt must be obtained as proof of disposal. Eastern Electric sends a manifest in subsequent mail within 35 days of waste pick-up and must also send a certificate of disposal within 30 days after the actual disposal date.

4.7.5 No annual report to the Department of Ecology is required because the level of PCBs is considered so high as to fall outside of the state's responsibility to monitor. At such levels the federal government regulates the disposal under TSCA. For this reason, it also does not fall within the federal requirement for RCRA governed waste disposal within 90 days. Eastern Electric is responsible for filing appropriate reports. TSCA

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regulations require that manifests and certificates of disposal be kept on file for a minimum of 3 years.

## 5. Reports

5.1 Disposal Paperwork

5.1.1 Our current vendor produces all of the required paperwork and insures all of the appropriate container markings (stickers, etc.) are in place prior to shipment. Since Laucks' waste streams are consistent, our vendors already have the information required to properly fill out the paperwork and Hazardous Waste stickers for all but the occasional labpack.

5.1.1.1 The paperwork includes the manifests, land disposal restriction forms and other shipping paperwork. Thus the only requirements of the laboratory are to insure the paperwork is accurate and to sign the appropriate forms.

5.1.2 After the waste has been transported to the disposal or accumulation facility, a signed manifest is returned to the laboratory. This is kept with the permanent record.

5.1.3 All certificates of disposal later provided by the disposal vendor are also associated with any waste shipment and kept with the permanent record.

5.1.4 All records are retained for at least 5 years from the date of shipment of the waste.

5.2 Annual Reporting Requirements

5.2.1 The laboratory must file an annual report with the Washington Department of Ecology (WDOE) for legal and tax purposes. This report is due on March 1 each year. Reports are filed for both the 940 and the 921 facilities (both EPA ID numbers). All waste transported from the 921 facility will be numbered WAD981762024 and all waste transported from the 940 facility will be numbered WAD027446608.

5.2.1.1 The only exception to this reporting requirement is the reporting of the PCB waste oil or soil with high enough levels of PCBs (>500 ppm) that they qualify as federally regulated TSCA waste and thus not reported to the WDOE.

5.2.2 The format of this report is defined by WDOE. In the past, WDOE provided books to the laboratory several months in advance of the due date with all of the required information. Currently, Laucks submits these through the online version of "Turbowaste" directly to the WDOE website. The instruction books are available online as well. We expect to continue using the web-based method of reporting for all future reports. Details of this report are not provided in this SOP.

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5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update by September 1 of each year. The pollution prevention plan is for the entire laboratory operation and as such, Laucks two sites are considered an interrelated facility with an ID number of IRF578002315. This report has been somewhat standardized since the last 5-year pollution prevention plan and it, too is now filed electronically via email. Details of this report are not part of this SOP.

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## Appendix I

### Hazardous Waste Sticker

See directions in this SOP for proper filling out of this sticker.

PROPER D.O.T. SHIPPING NAME HAZARDOUS WASTE, LIQUID N.O.S. NA9189

**ORM-E**

**HAZARDOUS WASTE**

FEDERAL LAW PROHIBITS IMPROPER DISPOSAL

IF FOUND, CONTACT THE NEAREST POLICE, OR
PUBLIC SAFETY AUTHORITY, OR THE
U.S. ENVIRONMENTAL PROTECTION AGENCY

GENERATOR INFORMATION:

NAME_____

ADDRESS_____

CITY_____ STATE_____ ZIP_____

EPA ID NO._____ EPA WASTE NO._____

ACCUMULATION START DATE_____ MANIFEST DOCUMENT NO._____

**HANDLE WITH CARE!**

CONTAINS HAZARDOUS OR TOXIC WASTES

STYLE WM-10

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**Appendix II**

**Example Hazard Class Stickers**

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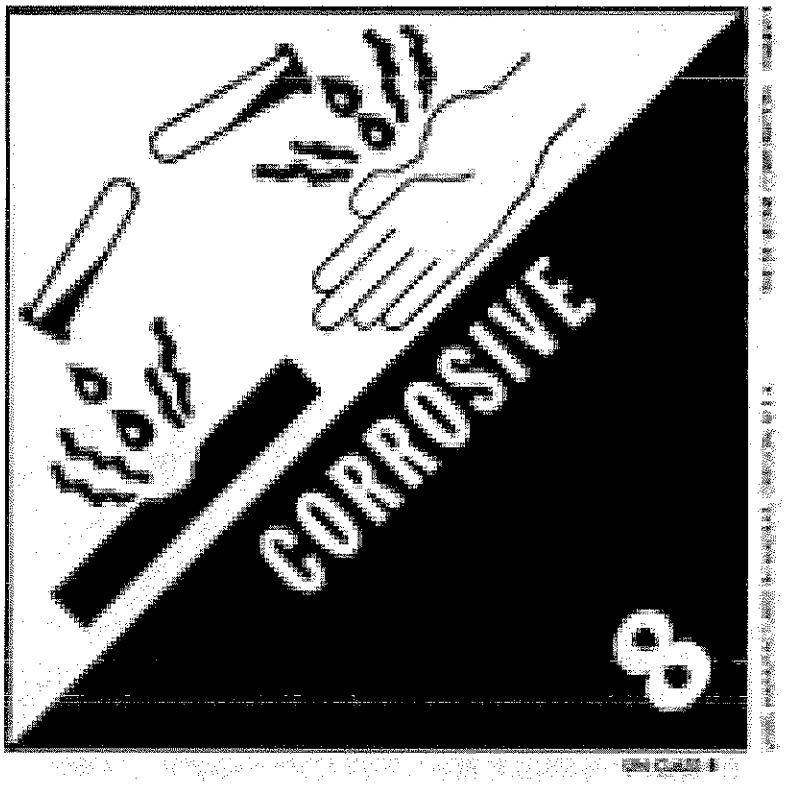

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## Appendix III

## A Quick Primer on Hazardous Waste Management

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## A Quick Primer on Hazardous Waste Management

Laucks is concerned and is indeed obligated by Washington State, to properly manage hazardous waste generated by the laboratory. This document is a quick overview for staff outlining the processes of which individuals should be aware. Everything is outlined in greater detail in Laucks SOP LTL-2001 *Waste segregation and Disposal*.

We currently manage 7 recurring hazardous waste streams at our 940 site and 4 at our 921 site. All individuals should be aware of the need to properly manage hazardous waste and those that need to know more should consult the aforementioned SOP. Any waste not covered in this document or in the SOP or which is not a regularly recurring waste stream must be discussed with QA for proper disposal.

**940 Site:**

- Solvent Waste – ALL solvent waste is collected in the 30 gallon drum in the solvent locker clearly labeled "Hazardous Waste". Solvent is collected in satellite containers located under the organic standards hood and in the waste solvent containers from the HPLC. Waste solvent should not be accumulated anywhere else without permission from QA. All other areas where solvent might be used should be immediately transferring the solvent to the 30 gallon container. Solvent is not to be dumped into any drum until it has been properly labeled.

- COD Waste – This is waste from the analysis for Chemical Oxygen Demand and is not only acidic but contains high concentrations of several metals, including Ag, $Cr^{+6}$ and Hg. It is collected in a clearly labeled 5 gallon carboy underneath the sink where CODs are analyzed. There should be no other collection point. NO OTHER WASTE SHOULD BE PUT INTO THIS CONTAINER WITHOUT THE EXPRESS PERMISSION OF QA. When the container is filled to 2 inches from the top, it should be tightly capped, the current date marked under the "accumulation start date" on the Hazardous Waste label, and the carboy immediately transferred to the acid storage locker. A new 5 gallon container should be started and QA should be contacted for the required labeling. Waste is not to be dumped into any carboy until it has been properly labeled.

- Soil – Waste soil samples are assumed to contain hazardous metals, specifically lead. Unless a soil is known to have been an agricultural soil or is known not to contain hazardous substances, it is treated as though it is hazardous. The soil samples are dumped into a 55 gallon drum in the 940 warehouse. When the drum is full, it is sealed, the current date marked under the "accumulation start date" on the Hazardous Waste label, and QA notified in order to arrange transport to a disposal facility. A new drum will be labeled by QA. Soil is not to be dumped into any drum until it has been properly labeled. All samples must be disposed of under chain-of-custody, i.e. scanned out of

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storage and into the heat-treatment oven or disposal drum. **Note: Off-shore and certain mainland U.S. soils must be heat treated prior to final disposal. Please read the full SOP for explicit instructions.**

- Extract Vials – Extract vials are collected in the covered, labeled 5 gallon bucket in the organic standards hood. When the bucket is full to the point that it can just be closed and sealed, it should be. The current date should then be marked as the start date on the Hazardous Waste label and QA contacted to arrange for disposal. A new 5 gallon container should be started and QA should be contacted for the required labeling. Waste is not to be dumped into any container until it has been properly labeled.

- Acidic Waste – Acidic waste containing significant amounts of metals should be disposed separately. QA should be consulted to make appropriate arrangements. Waste which is only acidic or has just trace amounts of metals MUST first be neutralized with bicarbonate and then can be poured down the drain into the domestic sewer. This waste, however, must be recorded in a logbook kept for that purpose. The SOP should be consulted for further information.

- Elemental Mercury & Debris Waste – Most of this waste is associated with sulfur removal of Pesticide/PCB samples. Other extraneous waste may be from broken Hg filled thermometers, Hg switches, etc. This waste is accumulated in the organics standards hood and is eventually lab-packed and disposed by our hazardous waste vendor.

- Fluorescent Lights – Fluorescent lights are considered "Universal Waste" and must never be disposed in the garbage. They must be collected and shipped to a recycling or hazardous waste disposal facility. These are collected in the boxes in which the replacement bulbs come, located in the 940 warehouse.

**921 Site:**

- Solvent Waste – ALL solvent waste is collected in the 55 gallon drum in the solvent locker clearly labeled "Hazardous Waste". Solvent may be collected in satellite containers located under some hoods but should NOT be allowed to sit uncovered and any such containers must be dumped into the 55 gallon container each night before the last analyst leaves. Solvent is not to be dumped into any drum until it has been properly labeled. Labeling instructions are in the SOP and labels are located in a drawer underneath the PCB hood.

- Soil – Waste soil samples are assumed to contain hazardous metals, specifically lead. Unless a soil is known to have been an agricultural soil or is known not to contain hazardous substances, it is treated as though it is hazardous. The soil samples are dumped into a 55 gallon drum in the 921 warehouse. When the drum is full, it is sealed,

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the current date marked under the "accumulation start date" on the Hazardous Waste label, and QA notified in order to arrange transport to a disposal facility. A new drum will be labeled by QA. Soil is not to be dumped into any drum until it has been properly labeled. All samples must be disposed of under chain-of-custody, i.e. scanned out of storage and into the heat-treatment oven or disposal drum. **Note: Off-shore and certain mainland U.S. soils must be heat treated prior to final disposal. Please read the full SOP for explicit instructions.**

- Fluorescent Lights – Fluorescent lights are considered "Universal Waste" and must never be disposed in the garbage. They must be collected and shipped to a recycling or hazardous waste disposal facility. These are collected in the boxes in which the replacement bulbs come, located in the 940 warehouse.

- PCB soil waste – Samples with low PCB levels (<2 mg/kg) are disposed in the normal soil drum after having been heat treated, if necessary. Soils with higher levels of PCBs are disposed in a separate, marked drum (also in the 921 warehouse) which will eventually be incinerated in order to destroy all PCBs and which will also serve the USDA APHIS requirement to heat treat any foreign soils prior to disposal.

- PCB Oil Waste – This waste is collected in a 5 gallon pail located in a blue plastic half-drum in the 921 warehouse. Only oil known or suspected to contain PCBs should be placed in this pail. Since we rarely get these oils anymore, the use of this container should be very infrequent. When it is full, QA is contacted to arrange for disposal and a new pail.

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# Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081B/8082A by 3535A-SPE)

Method # LTL-3205

June 30, 2006

Revision Number: #0

Written by: _____ Date: 6.30.06

Reviewed by: _____ Date: 9/6/06

Approved by: _____ Date: 9/6/06

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1. **PURPOSE** - In this method, organochlorine pesticides and polychlorinated biphenyls are extracted at pH <2 with acetone/methylene chloride. Using solid-phase extraction (SPE), target analytes are eluted from the solid-phase media, dehydrated, concentrated and exchanged into hexane. If necessary, the pesticide/PCB extracts may be GPC cleaned prior to GC analysis. PCB only extracts are acid cleaned prior to GC analysis. This method also includes low concentration method.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **DEFINITIONS**

Method Blank – An analytical control consisting of all reagents and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

Method Blank Spike - An analytical control consisting of all reagents, target analytes, and surrogate standards that is carried throughout the entire analytical procedure.

Matrix Spike – Aliquot of a matrix spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method.

Surrogates – Compounds added to each blank, blank spike, sample, matrix spike and spike duplicate to evaluate analytical efficiency by measuring recovery.

4. **REAGENTS**

4.1. All reagents shall be of AR grade or better.

4.2. All solvents shall be distilled in glass unless otherwise indicated.

4.3. The following special reagents shall be prepared:

4.3.1. 1:1 Sulfuric Acid - measure equal volumes of concentrated sulfuric acid and DIW. Note: Observe proper acid preparation techniques.

4.3.2. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

4.3.3. Surrogate solution prepared in acetone:

| | |
|---|---|
| 2, 4, 5, 6-Tetrachloro-m-xylene | 1.0 µg/mL |
| Decachlorobiphenyl | 1.0 µg/mL |

4.3.4. Pesticide matrix spiking solution prepared in acetone:

| | |
|---|---|
| 4,4'-DDD | 2.0 µg/mL |
| 4,4'-DDE | 2.0 µg/mL |
| 4,4'-DDT | 2.0 µg/mL |
| Aldrin | 1.0 µg/mL |
| alpha-BHC | 1.0 µg/mL |
| alpha-Chlordane | 1.0 µg/mL |

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| | |
|---|---|
| beta-BHC | 1.0 µg/mL |
| delta-BHC | 1.0 µg/mL |
| Dieldrin | 2.0 µg/mL |
| Endosulfan I | 1.0 µg/mL |
| Endosulfan II | 2.0 µg/mL |
| Endosulfan sulfate | 2.0 µg/mL |
| Endrin | 2.0 µg/mL |
| Endrin aldehyde | 2.0 µg/mL |
| Endrin Ketone | 2.0 µg/mL |
| gamma-BHC | 1.0 µg/mL |
| gamma-Chlordane | 1.0 µg/mL |
| Heptachlor | 1.0 µg/mL |
| Heptachlor epoxide | 1.0 µg/mL |
| Methoxychlor | 10 µg/mL |

4.3.5. PCB (AR1660) matrix spiking solution prepared in acetone:

| | |
|---|---|
| Aroclor 1016 | 25 µg/mL |
| Aroclor 1260 | 25 µg/mL |

Additional matrix spiking solutions such as Toxaphene, Chlordane and other Aroclors available on request.

## 5. EQUIPMENT

5.1. Six-port manifold station with two stage vacuum pump, capable of maintaining a vacuum of approx. 26 inches of mercury.

5.2. Waste liquid collection container.

5.3. Enviro-Clean Universal Extraction Cartridge – C18 (UCT)

5.4. Organomations Assoc., Inc. - N-EVAP, (Nitrogen Evaporator), Model 112

5.5. Vortex mixer

5.6. Standard laboratory glassware to include:

5.6.1. 40 mL collection vials (VOA)

5.6.2. K-D apparatus: 250 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

5.7. All glassware to be rinsed as follows, prior to use:

5.7.1. Technical grade acetone (if the glassware is wet).

5.7.2. Triple rinsed with methylene chloride.

5.8. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

## 6. SAMPLE COLLECTION & PRESERVATION

– Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ± 2°C. Water samples must be extracted within 7 days of collection.

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7. **POLLUTION PREVENTION & WASTE MANAGEMENT** – Sample extracts, standards and solvent rinses are disposed of by depositing them in the hazardous waste container located in the solvent locker. Waste segregation and disposal from the point of collection is further covered in LTL-2001.

8. **PERSONNEL QUALIFICATIONS** – This method is restricted to use by, or under the supervision of, experienced chemist/extractionists who have demonstrated the ability to perform the described extraction procedures and techniques.

9 **QUALITY CONTROL** - The normal level of quality control will consist of blank, blank spike, matrix spike and matrix spike duplicate (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

At least once every two years, performance of Toxaphene, Chlordane and all Aroclors will be demonstrated with a blank spike, matrix spike and matrix spike duplicate (MS and MSD).

10. **METHODOLOGY**

10.1. Sample Extraction

10.1.1. Bring sample to room temperature. Mix sample thoroughly in its original container and place labeling tape at sample level. Determine if the sample has suspended solids, which will require the use of a filter aid, filtering or centrifuging the entire sample. Mark meniscus of sample on the tape. Prepare label for all bottles. Following extraction, fill sample container to the mark on tape with water and measure volume of sample using a calibrated 1000 mL cylinder.

10.1.2. Prepare two additional aliquots of one sample, randomly selected or client designated, if quality control is required. Prepare separate MS/MSD for Pesticides and PCB.

10.1.3. Prepare a blank and blank spike with 1000 mL DIW in a clean one liter amber bottle. Prepare separate blank spike for Pesticides and PCB.

10.1.4. Pipet 200 µl surrogate solution into each sample bottle.
(Low Concentration: 20 µl surrogate solution)

10.1.5. Pipet 200 µl matrix spiking solution into MS/MSD and blank spike bottle.
(Low Concentration: 20 µl spiking solution)

10.1.6. Adjust pH to <2.

10.1.7. Soak cartridge for 1.5 minutes with 10 mL of methylene chloride, making sure that it does not go dry. Add additional as necessary.

10.1.8. Turn on vacuum and pull methylene chloride through cartridge until dry. Turn vacuum off.

10.1.9. Repeat 10.1.7. with acetone. Turn on vacuum and pull acetone through cartridge until dry. Turn vacuum off

10.1.10. Repeat 10.1.9. with methanol and pull through a small amount. After 1.5 minutes, turn on vacuum and pull remaining methanol through cartridge until just before cartridge goes dry. Turn vacuum off

10.1.11. Repeat 10.1.10. with 20 mL DIW. Turn on vacuum and pull DIW through cartridge.

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10.1.12. Just before cartridge goes dry, transfer label to port and invert sample bottle into the reservoir under full vacuum. After sample has gone through, dry cartridge at full vacuum for 10 minutes.

10.1.13. While cartridge is drying, remove sample bottle and rinse bottle with 10 mL acetone.

10.1.14. Following drying time, with vacuum off, place 40 mL collection vial into port under the cartridge. Add acetone from sample bottle to cartridge.

10.1.15. Rinse sample bottle with 10 mL methylene chloride and add to cartridge. Pull small amount through cartridge. Let soak for 1.5 minute.

10.1.16. Pull remaining solvent through cartridge until just before cartridge goes dry and repeat 10.1.15.

10.1.17 Turn off vacuum, remove vial, attach label and cap.

10.2. Solvent Dehydration

10.2.1. Prepare a stainless steel funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

10.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

10.2.3. Pass the extract from 10.1.17. through the funnel and collect in an assembled K-D apparatus.

10.2.4. Rinse the collection vial with several 10 ml methylene chloride rinses.

10.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

10.3. Solvent evaporation

10.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

10.3.2. Immerse tip of the ampule into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 8 ml. Regulate the evaporation time to take approx. 45 minutes.

10.3.3. Reduce the volume to 2-3 mL, and exchange into hexane as follows:

10.3.3.1. Add 2-3 mL hexane through the top of the Snyder column while the ampule is still immersed in the water bath. Reduce to 2-3 mL.

10.3.3.2. Repeat 10.3.3.1. two additional times.

10.3.4. Remove ampule from the water bath and cool to room temperature.

10.3.5. Rinse joint with hexane and remove the Snyder column. Allow rinse solvent to drain into ampule.

10.3.6. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of hexane.

10.3.7. Transfer extract to a 16 x 100 mm culture tube, and adjust to 10.0 mL final volume (as compared to a measured volume) with hexane. (Low concentration: 1.0 mL final volume.)

NOTE: If necessary, the pesticide/PCB extracts may be GPC (3640C) cleaned prior to GC analysis (see LTL-3692). All PCB only extracts must be sulfuric acid cleaned (3665A)

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10.4. Sulfuric Acid Cleanup (PCBs only) (3665A)

10.4.1. Add 2.0 mL concentrated sulfuric acid and vortex for 30-60 seconds. (Use 1.0 mL acid for low concentration)

10.4.2. Let stand for a few minutes to allow layers to separate. May be centrifuged.

10.4.3. Deliver extract in culture tube to 940.

10.4.4. Complete all necessary paperwork and bench sheet. Bench sheet should include bottle #, extraction solvent and final solvent lot #s, sodium sulfate lot #, cleanup method # if indicated, start and completion (to 940) date and time and extract location. Place folder in GC room extraction folder box. The file folder color will be purple and the blank name will be ____GPXWL_. PCB only will be a orange folder and the blank name will be ___GPB.WL_.

11. **TROUBLESHOOTING -** Please refer to Organic Extractions Troubleshooting Manual for detailed instructions on troubleshooting.

12. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

8081B (2001), 8082A (2001), 3535A (1998).

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# Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Soil (8081B/8082A by 3545A)

Method # LTL-3303

July 11, 2006

Revision Number: #5

Written by: B. O'Quiee Date: 7-11-06

Reviewed by: Harry Romberg Date: 7/20/06

Approved by: Hugh Prentice Date: 7/26/06

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1. **PURPOSE** - In this method organochlorine pesticides and polychlorinated biphenyls are extracted from neutral soil with a mixture of methylene chloride and acetone. The extract is dehydrated and concentrated in a Kuderna-Danish (K-D) apparatus. Pesticide/PCB extracts are GPC cleaned prior to analysis by GC. The PCB only extracts are sulfuric acid cleaned prior to analysis by GC.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples, by their very nature, may contain significant levels of hazardous materials. It is incumbent on each analyst to exercise due care and caution executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **DEFINITIONS**

Method Blank – An analytical control consisting of all reagents and surrogate standards that is carried throughout the entire analytical procedure. The method blank is used to define the level of laboratory, background, and reagent contamination.

Method Blank Spike - An analytical control consisting of all reagents, target analytes, and surrogate standards that is carried throughout the entire analytical procedure.

Matrix Spike – Aliquot of a matrix spiked with known quantities of specific compounds and subjected to the entire analytical procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.

Matrix Spike Duplicate – A second aliquot of the same matrix as the matrix spike that is spiked in order to determine the precision of the method.

Surrogates – Compounds added to each blank, blank spike, sample, matrix spike and spike duplicate to evaluate analytical efficiency by measuring recovery.

4 **REAGENTS**

4.1. All reagents are to be AR grade or better.

4.2. All solvents are to be distilled in glass, unless otherwise noted.

4.3. The following special reagents should be prepared:

4.3.1. 1:1 methylene chloride/acetone (v/v)- prepared by mixing equal volumes of the solvents.

4.3.2. Anhydrous sodium sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

4.3.3. Concentrated Sulfuric Acid

4.3.4. Accusand – prepared by soxhleting for 8 hours in methylene chloride to remove color, followed by muffling for four hours at 400°C.

4.3.5. Surrogate solution prepared in acetone:

| | |
|---|---|
| 2,4,5,6-Tetrachloro-m-xylene | 1.0 µg/mL |
| Decachlorobiphenyl | 1.0 µg/mL |

4.3.6. Pesticide matrix spiking solution prepared in acetone:

| | |
|---|---|
| 4,4'-DDD | 2.0 µg/mL |
| 4,4'-DDE | 2.0 µg/mL |
| 4,4'-DDT | 2.0 µg/mL |

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| | |
|---|---|
| Aldrin | 1.0 µg/mL |
| alpha-BHC | 1.0 µg/mL |
| alpha-Chlordane | 1.0 µg/mL |
| beta-BHC | 1.0 µg/mL |
| delta-BHC | 1.0 µg/mL |
| Dieldrin | 2.0 µg/mL |
| Endosulfan I | 1.0 µg/mL |
| Endosulfan II | 2.0 µg/mL |
| Endosulfan sulfate | 2.0 µg/mL |
| Endrin | 2.0 µg/mL |
| Endrin aldehyde | 2.0 µg/mL |
| Endrin Ketone | 2.0 µg/mL |
| gamma-BHC | 1.0 µg/mL |
| gamma-Chlordane | 1.0 µg/mL |
| Heptachlor | 1.0 µg/mL |
| Heptachlor epoxide | 1.0 µg/mL |
| Methoxychlor | 10 µg/mL |

4.3.7. PCB (AR1660) matrix spiking solution prepared in acetone:

| | |
|---|---|
| Aroclor 1016 | 25 µg/mL |
| Aroclor 1260 | 25 µg/mL |

Additional matrix spiking solutions such as Toxaphene, Chlordane and other Aroclors available on request.

## 5. EQUIPMENT

5.1. Dionex Accelerated Solvent Extractor - Model ASE 200 - Maintain per manufacturer's instructions.

5.1.1. 33 mL sample cells

5.1.2. Cellulose filters

5.2. Analytical Biochemical Laboratories, Inc. (ABC) Model 1002B or Model 1000 Gel-Permeation Chromatography (GPC)

5.3. Organomations Assoc., Inc. - N-EVAP (nitrogen evaporator), Model 112

5.4. Standard laboratory glassware to include:

5.4.1. 8 ounce extraction bottles

5.4.2. Small filling funnels

5.4.3. 60 mL collection vials

5.4.4. K-D apparatus: 250 mL K-D flask, 10 mL or 25 mL ampule and three-ball Snyder column.

5.5. All glassware and sample cells to be rinsed as follows, prior to use:

5.5.1. Technical grade acetone (if the glassware is wet).

5.5.2. Triple rinsed with methylene chloride.

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5.6. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

6. **SAMPLE COLLECTION & PRESERVATION** – Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C ± 2°C. Soil samples must be extracted within 14 days of collection.

7. **POLLUTION PREVENTION & WASTE MANAGEMENT** – Sample extracts, standards and solvent rinses are disposed of by depositing them in the hazardous waste container located in the solvent locker. Waste segregation and disposal from the point of collection is further covered in LTL-2001.

8. **PERSONNEL QUALIFICATIONS** – This method is restricted to use by, or under the supervision of, experienced chemist/extractionists who have demonstrated the ability to perform the described extraction procedures and techniques.

9. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

At least once every two years, performance of Toxaphene, Chlordane and all Aroclors, excluding 1016/1260, will be demonstrated with a blank spike, matrix spike and matrix spike duplicate (MS and MSD).

10. **METHODOLOGY**

10.1. Sample Extraction

10.1.1. Mix sample thoroughly in its original container if there is space available otherwise mix in solvent rinsed aluminum tray.

10.1.2. Weigh 15.0 grams of soil (wet weight) into a extraction bottle.

10.1.3. Prepare two additional aliquots of one sample, randomly selected or client designated, if quality control is required. Prepare separate MS/MSD for Pesticides and PCB.

10.1.4. Add 10 grams sodium sulfate and mix well to give the soil a sandy texture.

10.1.5. Insert cellulose filter into bottom of sample cell.

10.1.6. Using a small filling funnel, add approximately one gram sand to bottom of sample cell

10.1.7. Transfer sample to cell.

10.1.8. Prepare a blank and a blank spike with sodium sulfate. Prepare separate blank spike for Pesticides and PCB.

10.1.9. Pipet 100 µl surrogate solution to each cell.

10.1.10. Pipet 100 µl matrix spiking solution to each QC cell.

10.1.11. Fill cell to within 1 cm of top with sand or sodium sulfate and cap.

10.1.12. Fill solvent reservoir with 1:1 methylene chloride/acetone.

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10.1.13. Align trays and start.

10.2. Solvent dehydration

10.2.1. Prepare a stainless steel funnel by plugging with glass wool, and filling 1/4 full with sodium sulfate.

10.2.2. Pre-rinse the sodium sulfate by passing 40 mL methylene chloride through the prepared funnel.

10.2.3. Pass the extract from 10.1.13. through the funnel and collect in an assembled K-D apparatus.

10.2.4. Rinse the collection vessel with several 10 mL methylene chloride rinses.

10.2.5. Rinse the sodium sulfate with 25 mL of methylene chloride.

10.3. Solvent evaporation

10.3.1. Assemble the full K-D apparatus with a Snyder column prewet with 2-3 ml of methylene chloride.

10.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90°C, with a long ampule immersed to a depth of 12 mL. Regulate the evaporation time to take approx. 30 minutes.

10.3.3. Reduce the volume to 4-5 mL, remove the apparatus from the water bath and cool to room temperature.

NOTE: PCB Only: Reduce the volume to 4-5 mL and proceed to solvent exchange, 10.3.8.1. Bring to final volume of 5.0 mL in hexane. The solvent exchange step must be tested. Take 1 mL extract, 1 ml sulfuric acid and vortex. Check for volume changes between layers. If exchange is complete, proceed to 10.4.

10.3.4. Rinse joint and remove Snyder column. Allow rinse solvent to drain into ampule.

10.3.5. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of solvent.

10.3.6. Pesticides/PCB: Reduce the extract volume to less than 8 mL on a nitrogen blowdown. Transfer extract to a 16 x 100 mm culture tube, and adjust to 10.0 mL intermediate volume (as compared to a measured volume) with methylene chloride.

10.3.7. At this point the extract will be GPC cleaned (3640A) -- see Method # LTL-3692.

10.3.8. K-D the GPC cleaned extract to 4-5 mL and exchange into hexane as follows:

10.3.8.1. Add 2-3 mL hexane through the top of the Snyder column while the ampule is still immersed in the water bath. Reduce to 4-5 mL.

10.3.8.2. Repeat 10.3.8.1. two additional times.

10.3.9. Remove the apparatus from the water bath and cool to room temperature.

10.3.10. Rinse joint with hexane and remove Snyder column. Allow rinse solvent to drain into ampule.

10.3.11. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of hexane.

10.3.12. Reduce the extract to less than 2 mL in a nitrogen blowdown. Transfer extract to a 4.0 mL vial and adjust to a final volume of 2.5 mL (as compared to a measured volume) in hexane. Proceed to 10.4.4. PCB only final volume will be 5.0 mL in a 16 x 100 mm culture tube.

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10.4. Sulfuric Acid Cleanup (3665A) (PCBs only)

10.4.1. Add 2.0 mL concentrated sulfuric acid and vortex for 30 - 60 seconds.

10.4.2. Let stand for a few minutes to allow layers to separate. May be centrifuged.

10.4.3. Label the extract and deliver to 940.

10.4.4. Complete all necessary paperwork and bench sheet. . Bench sheet should include bottle #, extraction solvent and final solvent lot #s, sodium sulfate lot #, cleanup method #, GPC calibration date, start and completion (to 940) date and time and extract location. Place folder in GC room extraction folder box. The file folder color will be purple and the blank name will be ____GPX.SL_. PCB only will be a orange folder and the blank name will be ___GPB.SL_.

11. **TROUBLESHOOTING -** Please refer to Organic Extractions Troubleshooting Manual for detailed instructions on troubleshooting.

12. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846

8081B (2001), 8082A (2001), 3545A (1998), 3640A (1994), 3665A (1996)

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

Revision history:

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Written by: Pam Johnson Date: 5/21/07
Pam Johnson, Sample Control

Approved by: Hugh Prentice Date: 5/21/07
Hugh Prentice, Laboratory Director

Approved by: Harry Romberg Date: 5/21/07
Harry Romberg, QA Officer

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## 1. INTRODUCTION AND SCOPE

1.1 Introduction

1.1.1 This procedure is a description of sample receipt, sample log-in, and sample tracking when samples are logged into the laboratory's Laboratory Information Management System (LIMS). The collection of programs and procedures, which comprise the LIMS, is called "iLIMS." References made to iLIMS in this SOP are references to this collection of programs and procedures.

1.1.2 Sample entry must be performed in a timely fashion to allow tests with short holding times to be started immediately. Accuracy in the recording of sample IDs, in labeling samples with lab numbers, and in checking for consistency of all records is of utmost importance. All errors made at sample log-in, particularly sample ID's and test requirements, can cascade forward into all other subsequent analyses so attention to detail at this stage is critical.

1.2 Scope

1.2.1 All samples received by the laboratory are logged using the following procedures.

## 2. EQUIPMENT LIST

Lab coat
Safety glasses
Disposable gloves
Respirator, dust mask
3M desk cleaner, broom, dustpan, mop
Spatula
Label printer
PC work station linked to iLIMS
Barcode scanner
Fume Hood

## 3. SAFETY PRECAUTIONS

3.1 Sample Handling

3.1.1 Samples received at the laboratory can potentially be contaminated with toxic materials. Reasonable caution must be exercised at all times when handling these samples. Such precautions include wearing a lab coat at all times, wearing safety glasses, using gloves, using a hood (located in sample receiving) to perform operations when necessary (strong odors present, etc.), and wearing a respirator or dust mask if fumes or dust are generated.

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3.1.2 Cleanliness and neatness are of utmost importance. All spills and condensation from wet sample containers must be cleaned up immediately. This will help to alleviate accidental sample breakage and protect others from possible contact with contaminated work areas.

3.1.3 When wearing gloves, be certain to remove them when opening the cooler or lab doors and when answering the phone. The gloves, which protect the sample custodian from contamination, may transfer contamination to these objects. Other persons may touch the doorknob or phone without glove protection and have the contamination transferred to their unprotected hands. Never put pens, paper clips, etc. in your mouth.

3.1.4 A dust mask is worn when pouring dry packing material such as vermiculite into the garbage.

3.1.5 The sample entry hood is always used when unpacking samples with fumes (i.e. this is done for all Phillip samples) or for AFCEE samples even if odors are not present. Once the coolers have been opened, if breakage and/or odors are not present, the samples and cooler may be moved to a non-hooded area.

## 4. OPERATION PROCEDURES

4.1 Sample Receipt

4.1.1 Samples may be received by client delivery, over the front counter, via FED/EX, UPS, courier services, by various airfreight and overnight delivery services, and by Greyhound. It is the responsibility of the sample custodian to ensure that samples received by any of these services are promptly logged in and work requests made to the laboratory.

4.1.2 A traffic report (TR) or a chain-of-custody (COC) is received with the sample set. The sample custodian or a designated representative shall sign, date and record the time on all accompanying forms (e.g., TR/COCs, packing lists and airbills), when applicable. If the client has delivered the samples by hand, verify the cooler contents and return a copy of the COC to the client. NOTE: Initials are not acceptable. For EPA record the following information on Form DC-1 (Appendix 2) at the time of sample receipt:

- Presence or absence and condition of custody seals on shipping and/or sample containers.
- Custody seal numbers, when present.
- Condition of the sample bottles.
- Presence or absence of airbill or airbill stickers.

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- Airbill or airbill sticker numbers.
- Presence or absence of Traffic Report/Chain of Custody Records (TR/COCs) or Packing Lists.
- Sample tags listed/not listed on TR/COCs.
- Presence or absence of Traffic Reports or Packing Lists.
- Presence or absence of cooler temperature indicator bottle.
- Cooler temperature.
- Date of receipt.
- Time of receipt.
- EPA sample numbers.
- Presence or absence of sample tags.
- Sample tag numbers.
- Assigned laboratory numbers.
- Samples delivered by hand; and
- Problems and discrepancies.

4.1.3 If complete verification of the cooler contents will occur later, then the COC is stamped and the stamped copy returned to the client. This stamp is reproduced in Appendix 1. Verification must take place within one working day of receipt.

4.1.4 All discrepancies between the Traffic Report/COC and the actual samples received are immediately reported to the client. This is done by the assigned project manager, or a designated representative in the event that the project manager is not available and are noted on the appropriate Sample Receipt Log. For the EPA discrepancies are noted on the Form DC-1 (Appendix 2). The assigned project manager or designated representative shall contact the Sample Management Office (SMO) to resolve problems and discrepancies including, but not limited to absent documents, conflicting information, air bubbles in voa vials, absent or broken custody seals, absent temperature indicator bottle, and unsatisfactory sample condition (e.g., leaking sample container). The project manager shall record the resolution of all problems and discrepancies communicated through SMO. The Sample Receipt Log (Appendix 3) is for all log-in procedures.

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4.1.5 Put on gloves, open the coolers (in the hood if necessary), and note whether custody seals are present and, if so, intact. Record custody seal numbers, when present, record the date the sample container was sealed and any additional information indicated on the custody seals. If there is a question about the integrity of the custody seals, make a note on the EPA Form DC-1 (Appendix 2), or the Sample Receipt Log (Appendix 3); the client must be informed.

4.1.6 After the coolers are opened, determine whether there are soil or water samples in the coolers. Typically there will be a number of sample bottles for each sample if we received waters. Soils will have only a small number of containers per sample.

4.1.7 Visually check the contents of the opened cooler for obvious damage or broken sample containers. Note any breakage on the appropriate Sample Receipt Log. For EPA note breakage on Form DC-1 (Appendix 2).

4.1.8 For any program (such as EPA, AFCEE, NFESC, or Army Corps) or CLP samples, either the enclosed temperature blank or at least 3 separate containers taken randomly from different locations in EACH cooler must be checked for temperature with the infrared thermometer. If the enclosed temperature blank exceeds 4°C ± 2°C then three additional sample containers should be checked. The temperatures are recorded on the Sample Receipt Log (Appendix 3). If any samples exceed the range of 4°C ± 2°C the Project Manager must be notified, and the client should be contacted as soon as possible. In most cases, this should be done **in writing** (preferably FAX or email) by the appropriate project manager. A copy of the communication from the Sample Receipt Log must be kept with the COC in the SDG file.

4.1.9 If samples for GC/MS volatiles analysis are received in unpreserved vials, or if they are received near the end of the established holding time, the volatiles staff should be notified immediately via email.

4.1.10 Remove all bottles from the cooler and put on the bench.

4.1.11 As you are placing the samples on the bench, they should be screened for radioactivity by passing the sample containers near the detector window of a Geiger counter. Further details can found in Laucks SOP on Screening of Radioactivity in Samples.

4.1.12 Line up the bottles in some kind of order, if there is an apparent order. Various means of ordering samples are:

- COC order
- Client sample ID
- Date sampled

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- Time sampled

4.1.13 For samples consisting of multiple containers, place all containers together on the bench.

4.1.14 After all samples are arranged then check consistency between the COC and the sample labels for sample IDs, dates and times on each sample container. At this time VOA vials should also be checked for the presence of air bubbles. If bubbles are present it should be noted on the Sample Receipt Log Form (Appendix 3) if they are greater than or less than ¼". The appropriate Project Managers must be notified immediately and the client should be contacted as soon as possible if the air bubbles are greater than ¼".

4.1.15 Determine whether custody seals are present on the individual sample containers (jars and bottles). If present and intact, so note. If present and any seal is broken, so note. These notations must be made on the EPA form DC-1 (Appendix 2) or the Sample Receipt Log Form (Appendix 3).

4.1.16 All water sample bottles for CLP work must be checked for pH at the time of sample receipt. Pouring out some of the sample into a small plastic cup and then using pH paper to record the pH at time of receipt does this. Volatiles samples should **NOT** be checked. Volatiles analysts will do this after analysis. When better discrimination of pH is needed, narrow range pH paper should be used to confirm the pH (especially if the pH is within 1 pH unit of the required preservation limit for that sample). All pH measurements must be recorded on the Supplemental Sample Receipt Log (Appendix 3). If any samples exceed the pH requirements, the Project Manager must be notified, and the client should be contacted as soon as possible. For evidenciary purposes, this should be done **in writing** (preferably FAX or email) by the appropriate project manager. The samples with inappropriate pH are listed on Laucks Testing Lab pH log form (Appendix 5) for corrective action. Sample pH is adjusted appropriately and the volume **AND** type of preservative is recorded. After the correct preservation is complete, this form is given to the appropriate project manager for SDG filing.

4.1.17 Some samples are received at the lab and will need to be split and preserved for different analytes. To accommodate preservation requirements, these samples are recorded on the "Sample Split Sheet" (see Appendix 6). There is a specific cart located in sample entry where the samples are temporarily stored until splitting and preservation takes place. After the correct preservation is complete this form is given to the appropriate project manager for SDG filing.

4.1.18 All sample container marks (including ID's, dates and times) are then verified with each other and with the COC. This procedure is completed by noting whether all bottles from the same sample have the same ID and whether this ID is the same as on the COC. All

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discrepancies are noted on the Sample Receipt Log (Appendix 3) and reported to the client.

4.1.19 To determine if the sample(s) is (are) acceptable, compare the existing conditions with the criteria specified in (Appendix 7), "Required Containers/Volumes, Preservation Techniques and Maximum Holding Times for Environmental Analysis". All listed criteria must be met in order to qualify the sample(s) as "acceptable". If there are any problems with the sample(s) these must be documented in the Sample Receipt Log (Appendix 3). If any samples are not acceptable, the client must be contacted. In most cases, this should be done **in writing** (preferably FAX or email) by the appropriate project manager.

4.2 Sample Log-In

4.2.1 Determine whether a client record exists in the iLIMS database. If it does not, create a record. At a minimum, the client record will include:

- an alphanumeric client code (up to 12 digits)
- the client's full and accurate name, address, and point of contact
- the client's telephone number and/or FAX number
- the full and complete address for reports and invoices
- the purchase order/contract number if that number applies to all work the client may submit. (If the purchase order/contract number is specific to one sample submittal, by project etc.), then it would be project specific to the client code. Example (client name_project name).

4.2.2 An iLIMS SDG is started for the job through the iLIMS home page by clicking on the SAMPLES icon. An SDG for COA clients is typically identified by using 4 alphabetical codes specific to the client name and a 6-digit number, which is assigned by iLIMS at the time the SDG is initiated. The four letters represent portions of the client name, the first set of two digits represent the year, the second set of digits represent the month and the final two digits represent how many SDGs have been created for that client within that month. For instance, SDG NRTC060603 is the code for Nestle Regional Training Center, the year is 2006, the month is June and this was the third SDG created for that client within the month of June. This number will be used throughout the laboratory to track the job. An SDG for CLP is identified by using two or three alphabetical codes specific to the project name along with two digits indicating the number of SDGs within the project. For example, LTM01 is the code for the project "Long Term Monitoring" and "01" is the first SDG within that project name.

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4.2.3 The SDG is created at sample login. The first step is inspection of the shipping containers. Click the box next to (No Container Present) if samples were received without a shipping container. If a shipping container was received click on the "Login Container" button. A Sample Receipt Log" (Appendix 3) form will appear and need to be completed by answering all the questions on the form. To add an existing cooler click on the button "Add Existing Container" and another screen will appear. You can search for an existing cooler in several ways. You can search by the client name, the SDG, the container description or by the COC number. Click the refresh button for coolers to appear. Find the container you are searching for and click in the box to add the cooler.

4.2.4 The second step is selecting the SDG. If you know the client type the name in the box, (enter as much of the name as you know) then hit the enter key. A list of clients will appear in the box, select your client name from the list and click next. If you need to create a client you can do this by clicking in the circle next to "I need to create a client" fill in the name, system ID (if available), address, city, state and zip code, click next. After selecting the client, another box will appear and you will need to select a project for the SDG. Use the drop down list to select a project. An example of a project is "drinking water", or "storm water testing". After selecting the project you can choose to create a new SDG or add to an existing SDG. Select the existing SDG from the drop down list, then click next.

4.2.5 If you had to create a client you will need to "Add Tests" in step 3. Otherwise move to Step 4 – Login Samples.

4.3 Special identification for samples received from overseas

4.3.1 Samples received from outside the continental United States such as Pacific islands (including Hawaii, but not the Alaska Aleutian Islands) or even soil from Vancouver Island in Canada must be heat-treated prior to disposal. These samples must be marked with a red dot on the label or lid during sample entry to ensure proper disposal will occur. In addition to marking the samples with red dots, also place red dots on all internal COCs associated with the sample. If samples are subcontracted to another lab include a soil permit for shipment of these samples, that lab must also be notified that the samples will need to be heat-treated prior to disposal.

4.3.2 Prior to sample disposal, these soil samples must be treated with dry heat by placing them in a 250 °F (121 °C) oven for at least 2 hours. Laucks' practice is to remove the caps (place them in the oven separately), place soil jars containing soil in the oven and heat them for 4 hours at 125 °C.

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4.4 Special documentation procedures for CLP samples

4.4.1 Completion of the CLP Sample Receipt Log, and the Supplemental Sample Receipt Log

4.4.2 The Sample Receipt Log and the Supplemental Sample Receipt Log are CLP-specific sample login sheets. For each cooler received a CLP Sample Receipt Log and a Supplemental Sample Receipt Log must be completed. Copies of these forms may be found in (Appendix 3).

4.4.3 Complete the Non-Conformance checklist. If there is a problem with the custody seals, chain of custody records, or agreement between the custody records, the client must be contacted. In this case, this should be done **in writing** (preferably FAX) by the appropriate project manager. A copy of the communication must be kept with the COC in the SDG file.

~~4.4.4~~ Since the extractable fractions will be transferred to the extractions lab, the samples are scanned onto shelf 8C in the W01 walk-in cooler (extractions hold shelf) pending pick-up by extractions personnel.

4.5 Assignment of lab quality control samples

4.5.1 The client may choose to designate which samples are to be analyzed as a matrix spike/matrix spike duplicate. This means that the sample preparations and the VOA departments cannot self-assign QC samples until all samples from the SDG are received. It is the responsibility of the sample login person to notify the operations staff when a specified QC sample is received by marking it as a specified QC sample.

4.6 Sample storage

4.6.1 The following tests must be started very soon after receipt when performed on water samples. See (Appendix 8) for Checklist For Analyses With Short Holding Times.

| Test Type/Name | Holding Times |
|---|---|
| $NO_3$ – nitrate (300.0) | 48 hours |
| $NO_2$ – nitrite (300.0) | 48 hours |
| $NO_2$ – nitrite (354.1) | 48 hours |
| $Cr^{+6}$ - hexavalent chromium | 24 hours |
| $CO_2$ - carbon dioxide | Immediate |
| DO - dissolved oxygen | 24 hours |
| BOD - Biological Oxygen Demand | 48 hours |
| Chlor A – Chlorophyll A | 24 hours |
| Settleable Solids | Immediate |

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| | |
|---|---|
| Filtration for dissolved metals | Immediate |
| PH | Immediate |
| Microbiological tests | Various |
| Color | 48 hours |
| Turbidity | 48 hours |
| Sulfite | 24 hours |
| MBAS - Methylene Blue Active Substances | 7 days |
| Residual Chlorine | 24 hours |
| Ortho phosphorus/soluble reactive phosphate | 48 hours |
| Ferrous Iron | 24 hours |
| Sample Preservation/sample splitting | Immediate |

4.6.2 If samples arrive late in the day, which have holding time issues that will expire if the analysis is not started that day. The sample custodian must immediately take the containers to the work areas in which the tests will be performed and the primary person responsible for these tests notified that samples are here. A list with the name of the appropriate analyst is posted in the sample receiving area.

4.6.3 When micro samples are received, the micro department is notified immediately by phone.

4.7 Storage locations

| **Location** | **Description of contents** |
|---|---|
| VOA refrigerator | Soil and water volatiles, (GCMS C07, GC R04) |
| Shelf | Aqueous metals, oils, no cooling required |
| Inorganics cooler | COA small water jobs, (C05) |
| Walk-in cooler | All other soil and water *, W01 |
| Shelf 1D in the walk-in cooler are for COA soil/miscellaneous samples | All samples that are under internal COC. CLP samples are also stored in here. |

*See Section 4.4.4 for transfer of extractable aliquots to the extractions lab

4.7.1 Prior to putting bottles into any storage location, scan the location, then scan the bottles for storage. See (Appendix 4) for the Secure Storage Custody Log, Organic Extractions Custody Log.

4.8 Determination of tests

4.8.1 Project Managers create all client information and project determinations (tests) before samples are logged into the iLIMS database. This reduces the likelihood of sample entry

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personnel selecting the wrong determination for a specific sample container. If Laucks provided sample bottles for the client, the bottle order, the client COC, file notes, letters, client instructions are consulted as necessary to determine what tests are to be performed. If the tests can not be determined the Project Manager will contact the client.

4.8.2 Some soil samples will need to be shared between two or more departments. Place comments in the comment section under "sample notes notifying each department that only one container was submitted and that these containers must be shared. Identify all departments involved in the text of the comment and direct them to notify the next department via email that the sample is available when they are done with it. Sample entry may need to help with the sample login processes described below. In order of priority, the following areas will receive samples in this order:

4.8.3 If volatiles are requested, then the VOA departments will get the samples first (GC or GC/MS). When VOAs are done, they will do one of the following:

4.8.3.1 Notify extractions vial email that the sample is available and scan the sample into the 8C shelf in the walk-in refrigerator. The sample/samples will then be sent to the extraction lab. When extractions is done with the sample, they will notify inorganics that the sample is available and scan it into an available shelf in the walk-in refrigerator at 940.

4.9 Generation of internal Chain-of-Custody (COC)

4.9.1 All samples must be scanned to the analyst when they are removed from storage, and scanned back to the storage location when they are returned.

4.10 Sample breakage

4.10.1 All sample breakage whether in shipping or while handling in the lab, must be reported immediately to the Project Manager.

4.10.2 If the sample was water, clean thoroughly with disposable towels. Be very careful with broken glass so as to avoid cuts.

4.10.3 If the sample was soil, as much of the sample as possible is transferred to a new, clean jar using a spatula. Be certain not to pick up any sample, which has contacted the floor. Save the original label, if possible. Note on the log-in records that the sample was broken and transferred to a new container.

4.10.4 All dirty, disposable clean up materials, soil, broken glass, etc. are placed in a plastic garbage bag before being placed in the dumpster. All non-disposable clean up materials are washed after use.

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4.11 Special circumstances

4.11.1 Samples from some clients are logged into a monthly SDG. Some jobs extending over more than one sampling event may be entered under one SDG number. In that event, pay special attention to date of receipt. The Project Managers will review SDG/COCs for each date of sample receipt.

4.11.2 Other special circumstances may arise. If there are any questions, check with the Project Manager first.

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# APPENDIX 1

## Sample Receipt Verification Stamp

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# APPENDIX 2

## EPA Form DC-1

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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: | | | Corresponding | | |
| | | EPA Sample # | Sample Tag # | Assigned Lab # | Remarks: Condition of Sample Shipment, etc. |
| 1. Custody Seal(s) | Present/Absent* Intact/Broken | | | | |
| 2. Custody Seal Nos. | _____ _____ | | | | |
| 3. Traffic Reports/ Chain of (TR/COC) 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 Temperature | _____ | | | | |
| 10. Does information on TR/COC Records and sample tags agree? | Yes/No* | | | | |
| 11. Date Received at Laboratory | _____ | | | | |
| 12. Time Received | _____ | | | | |
| 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. |

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# APPENDIX 3

**CLP Sample Receipt Log**

**Supplemental Sample Receipt Log**

## LAUCKS TESTING LABORATORIES

**Login Shipping Container**
*Please note any discrepancies in the comments*

| | | |
|---|---|---|
| 1. | Please describe the container: *(e.g. blue cooler #1, FedEx Shipping Box)* | |
| 2. | What type of container is this? | Cooler |
| 3. | Taken: | |
| 4. | Transferred: | |
| 5. | Chain of Custody #: | |
| 6. | Logged In By: | Rachel Frank |
| 7. | Received On: | 10/04/2006 08:35 AM |
| 8. | Container Opened On: | 10/04/2006 08:35 AM |
| 9. | Did container come with a shipping slip? *(if yes then record carrier name and airbill number)* | ☑ Yes |
| 10. | Custody seals were? *(please describe)* | Intact<br>10/03/2006 08:35 AM |
| 11. | Were custody papers sealed and taped to container? | ☑ Yes |
| 12. | Were custody papers properly filled out? | ☑ Yes |
| 13. | Did you sign the custody papers? | ☑ Yes |
| 14. | Did you screen for radioactivity using the Geiger Counter? | ☐ Yes |
| 15. | If required, was enough cooling material present? | ☑ Yes |
| 16. | Describe type of packing in container: | |
| 17. | Were all bottles sealed in separate plastic bags? | ☑ Yes |
| 18. | Were all bottle lables complete(ID, date, time signature, preservative, etc)? | ☑ Yes |
| 19. | Did all bottle lables agree with custody papers? | ☑ Yes |
| 20. | Were correct containers used for the tests indicated? | ☑ Yes |
| 21. | Were correct preservatives added to samples? | ☑ Yes |
| 22. | Put any temperature readings here: | |

**Comments**

23.

Save

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**Supplemental Sample Receipt Log**
**Laucks Testing Laboratories**

**SDG:** CDVL061001
**Cooler:** AAD343
**Temperatures:**
**COC #:** 40198

| Sample | Bottle # | Bottle Description | pH | Bubbles |
|---|---|---|---|---|
| CDVL061001-003 | 0001 | 1000 mL juice, plastic | 7 | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0004 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0005 | 500 ml cylinder, poly, HNO3 | <2 | None |
| CDVL061001-009 | 0001 | 1000 mL juice, plastic | 7 | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0004 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0005 | 500 ml cylinder, poly, HNO3 | <2 | None |
| CDVL061001-012 | 0001 | 1000 mL juice, plastic | 7 | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0004 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0005 | 500 ml cylinder, poly, HNO3 | <2 | None |
| CDVL061001-014 | 0001 | 1000 mL juice, plastic | 7 | None |
| | 0002 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0003 | 40 ml OTWS, clear glass, H3PO4 | N/C | None |
| | 0004 | 500 ml cylinder, poly, H2SO4 | <2 | None |
| | 0005 | 500 ml cylinder, poly, HNO3 | <2 | None |

Allowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

| | |
|---|---|
| Temperature | Allowable temperature range is 4+/- 2 degrees Celsius |
| Acid Preserved pH | pH must be less than 2 |
| Base Preserved pH | pH must be greater than 12 |
| NC | Not Checked for pH |

*FORM LTL-PM-16.0*

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# APPENDIX 4

**Secure Storage Custody Log**

**Organic Extractions Custody Log**

laboratory information management system

ACCOUNTING | CONFIGURATION | SAMPLES | CLIENTS/PROJECTS | METHODS/ANALYTES | ANALYSIS | STANDARDS LOG

Logged in as **Pamela Johnson**
**120** minutes before system logs you out

HELP | USER MANUAL | LOG OUT

**View**
- > Open SDG
- Clients
- Bids
- Projects
- Pricebooks
- Bottle Orders
- Calls logged
- Invoices

**Tasks**
- Log a phone call

**Project**
- View Project
- Authorizations
- Offices/Contacts/Files

**SDG**
- View SDG
- Sample Review Tree
- Sample
- Confirmation Log
- Account History

## CLIENTS/PROJECTS **Sample Bottle**

Properties | Custody

| Moved By | On | Moved From | To |
|---|---|---|---|
| Amanda Pease | 09/12/2006 10:21 | <n/a> | Sample Login Area |
| Amanda Pease | 09/12/2006 11:32 | Sample Login Area | LW-5B |
| Jennifer Penner | 09/13/2006 08:39 | LW-5B | Jennifer Penner |
| Rachel Frank | 09/13/2006 14:59 | Jennifer Penner | LW-5B |

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laboratory information management system

ACCOUNTING | CONFIGURATION | SAMPLES | CLIENTS/PROJECTS | METHODS/ANALYTES | ANALYSIS | STANDARDS LOG

Logged in as **Pamela Johnson**
**120** minutes before system logs you out

HELP | USER MANUAL | LOG OUT

**View**
- > Open SDG
- Clients
- Bids
- Projects
- Pricebooks
- Bottle Orders
- Calls logged
- Invoices

**Tasks**
- Log a phone call

**Project**
- View Project
- Authorizations
- Offices/Contacts/Files

**SDG**
- View SDG
- Sample Review Tree
- Sample
- Confirmation Log
- Account History

## CLIENTS/PROJECTS **Sample Bottle**

Properties | **Custody**

| Moved By | On | Moved From | To |
|---|---|---|---|
| Amanda Pease | 09/12/2006 10:21 | <n/a> | Sample Login Area |
| Amanda Pease | 09/12/2006 10:59 | Sample Login Area | LW-8C |
| Vinh Ly | 09/12/2006 12:00 | LW-8C | EXW-B14 |
| Lita Suafoa | 09/14/2006 11:58 | EXW-B14 | Lita Suafoa |
| Lita Suafoa | 09/15/2006 08:31 | Lita Suafoa | Sample consumed - Ext. |

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# APPENDIX 5

**Laucks Testing Lab pH Log Form**

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**Laucks Testing Lab pH log Form**

Date_____

| Analyst | WO Number | Fractions | pH before | Volume Acid Added/Type | Resulting pH |
|---|---|---|---|---|---|
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# APPENDIX 6

**Laucks Testing Lab Sample Split Sheet**

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## Laucks Testing Lab Sample Split Sheet

| WO # | Sample # | WE HAVE | WE NEED | TOTAL | DISS | UNPRES | INTL & DATE | BOTTLE TYPES |
|---|---|---|---|---|---|---|---|---|
| | | | | | | | | Y = YELLOW |
| | | | | | | | | B = BLUE |
| | | | | | | | | C = CYANIDE |
| | | | | | | | | R = RED |
| | | | | | | | | O = O&G |
| | | | | | | | | P = PHENOL |
| | | | | | | | | S = SULFIDE |
| | | | | | | | | T = TOC |
| | | | | | | | | OTHER |
| | | | | | | | | |
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# APPENDIX 7

**Required Containers/Volumes/Preservation/Holding Times**

# REQUIRED CONTAINERS/VOLUMES, PRESERVATION TECHNIQUES AND MAXIMUM HOLDING TIMES FOR ENVIRONMENTAL ANALYSIS

## A. PRIORITY POLLUTANTS - Organics Analysis (Federal Register Vol. 72, No. 47, March 12, 2007)

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Purgeable Halocarbons | 3 - 40 ml containers | Glass, Teflon-lined Septum, 40 ml capacity | Cool, ≤6° C, no headspace; 0.008% $Na_2S_2O_3$ if $Cl_2$ present | 14 days, with preservation | Method 624, GC/MS |
| Purgeable Aromatic Hydrocarbons | 3 - 40 ml containers | Glass, Teflon-lined Septum, 40 ml capacity | Cool, ≤6° C, adjust pH to ≤ 2 HCl, no headspace; 0.008% $Na_2S_2O_3$ if $Cl_2$ present | 14 days, with preservation<br>7 days, if not preserved | Method 624, GC/MS |
| Acrolein and Acrylonitrile | 3 - 40 ml containers | Glass, Teflon-lined Septum, 40 ml capacity | Cool, ≤6° C, no headspace; 0.008% $Na_2S_2O_3$ if $Cl_2$ present<br>As above, and pH adjusted to pH 4-5. | 14 days<br>14 days | Method 624, GC/MS |
| Phenols | 1 liter | Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity | Cool, ≤6° C, no headspace; 0.008% $Na_2S_2O_3$ if $Cl_2$ present | 7 days until extraction; 40 days after extraction for analysis | Method 625, GC/MS |
| Pesticides (Organochlorine Pesticides, and PCB's | 1 liter | Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity | Cool, ≤6° C; pH 5-9 | 7 days until extraction; 40 days after extraction for analysis | Method 608, GC |
| Polynuclear Aromatic Hydrocarbons (PAHs) | 1 liter | Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity | Cool, ≤6° C; store in dark; $Na_2S_2O_3$ if $Cl_2$ present | 7 days until extraction; 40 days after extraction for analysis | Method 610, GC or Method 625, GC/MS |
| Base/Neutral and Acid Extractables | 1 liter | Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity | Cool, ≤6° C | 7 days until extraction; 40 days after extraction for analysis | Method 625, GC/MS |

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## B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition)

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Halogenated Volatile Organics | 3 - 40 ml containers for liquids<br>20 grams for solids | Glass, Teflon-lined Septum, 40 ml capacity<br>Above or Glass, 2-4 oz. Capacity, or Encores | Cool, 4° C, no headspace<br>Cool, 4° C, packed to avoid headspace | 14 days<br>14 days | Method 8260, GC/MS Purge-and-Trap Method 5030/5035 |
| Nonhalogenated Volatile Organics | 3- 40 ml containers for liquids<br>20 grams for solids | Glass, Teflon-lined Septum, 40 ml capacity<br>Above or Glass, 2-4 oz. capacity, or Encores | Cool, 4° C, adjust pH ≤ 2 with HCl, no headspace<br>Cool, 4° C, packed to avoid headspace | 14 days, with preservation<br>7 days, if not preserved<br>14 days | Method 8015 GC/FID Direct Injection or Purge-and-Trap, Method 5030/5035 or Method 8260, GC/MS Purge-and-Trap Method 5030/5035 |
| Aromatic Volatile Organics | 2 - 40 ml containers for liquids<br>20 grams for solids | Glass, Teflon-lined Septum, 40 ml capacity<br>Above or Glass, 2-4 oz. Capacity or Encores | Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid and adjust pH to ≤2 with HCl, no headspace<br>Cool, 4° C | 14 days with preservation<br>14 days | Method 8021, GC/PID Direct Injection or Purge-and-Trap, Method 5030/5035 Method 8260, GC/MS Purge-and-Trap Method 5030/5035 |
| Methane/Ethane/Ethylene (not SW 846) | 3 – 16.5 ml containers for liquids | Glass, Teflon-lined Septum, 16.5 ml capacity | Cool, 4° C, adjust pH 2 with HCl, no headspace | 14 days, with preservation | Method RSK 175, GC/FID |
| Phenols | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C, 35 mg $Na_2S_2O_3$ per ppm free chlorine per liter, adjust pH <2 with $H_2SO_4$<br>Cool 4° C | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8270 GC/MS |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Organochlorine Pesticides and PCBs | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C, adjust pH to 6-8 with $H_2SO_4$ or NaOH<br>Cool, 4° C | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8081/8082 GC/ECD |
| Polynuclear Aromatic Hydrocarbons (PAHs) | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8310 HPLC<br>or<br>Method 8270 GC/MS |
| Chlorinated Hydrocarbons | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8270 GC/MS |
| Organo-phosphorus Pesticides | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8141 GC/NPD or GC/NPD/ECD or Method 8270 GC/MS |
| Chlorinated Herbicides (i.e., 2,4-D and 2,4,5-TP) | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8151 Extraction and Esterification/GC-ECD |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Volatile Organics (VOAs) | 3- 40 ml containers for liquid sample<br>20 grams for solids | Glass, Teflon-lined Septum 40 ml capacity<br>As above or glass, 2-4 oz. or Encores | Cool, 4° C, acid preserved with HCl to pH < 2, no headspace<br>Cool, 4° C, no headspace or if solid packed to minimize headspace | 14 days<br>14 days | Method 8260 Purge-and-Trap GC/MS |
| Semi-Volatile Organics | Approximately 1 liter for liquid sample<br>Approximately 50 grams for sludge or solid sample | Glass, Teflon-lined cap | Cool, 4° C, | Extracted within 7 days and completely analyzed within 40 days<br>Extracted within 14 days and completely analyzed within 40 days | Method 8270 GC/MS |

## C. WASTE EVALUATION - GENERAL

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Toxicity Characteristic Leaching Procedure | Approximately 1 liter for liquid sample<br>Approximately 200 grams for solid sample | Glass, Teflon-lined cap | cool to 4°C ± 2°C prior to TCLP extraction<br>After extraction:<br>VOA - pH<2 with HCl<br>Metals - pH <2 with $HNO_3$<br>Semivolatiles, Pesticides/Herbicides - | VOA, Metals, Semivolatiles, Pesticides/Herbicides - 14 days until extraction. Follow analytical protocol for aqueous holding time or holding time from leachate preparation. | According to requested analysis |
| Reactivity | Approximately 100 ml for liquid sample<br>Approximately 50 grams for solid sample | ----- | None | not specified | Chapter 7 |

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## C. WASTE EVALUATION - GENERAL (continued)

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Ignitability | Approximately 200 ml or 50 grams | None specified | None | not specified | Method 1010 Pensky-Martens Closed-Cup Method<br>Method 1020 Setaflash Closed Cup Method |
| Corrosivity | 100 - 500 ml | Plastic | None | None | Method 1110 Corrosivity Toward Steel: Method 9040 or 9045 |
| California Assessment Manual CAM/STLC | Approximately 200 ml for liquid sample<br>Approximately 10 grams for solid sample | Plastic or Glass | No preservation for solid sample. Add $HNO_3$ to pH <2 for liquid sample | 28 days for Mercury 6 months for others | CA WET<br>See Methods for Metals Analysis |

## D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition)

| PARAMETER | MINIMUM VOLUME REQUIRED ** | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Mercury, Total/Dissolved<br>Mercury, Total | 100-200 ml for liquid sample<br>Approximately 5 grams for solid sample | Plastic or Glass | $HNO_3$ to pH <2 for total<br>Filter on site, $HNO_3$ to pH <2 for dissolved | 28 days | EPA 245.1 for water or EPA 7470A<br>EPA 7471A for sediment Cold Vapor Method |
| Metals, Total<br>Metals, Dissolved | 300 ml for liquid samples<br>Approximately 10 grams for solid samples | Plastic or Glass | $HNO_3$ to pH <2 for total<br>Filter on site, $HNO_3$ to pH <2 for dissolved | 6 months | ICP- 200.7 or 6010; ICP/MS 200.8 or 6020 |

**** For individual metals the aggregate minimum volume is determined by the number of discrete analytical methods not the sum of all the individual analytes.**

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## D. METALS ANALYSIS (EPA Methods for Chemical Analysis of Water and Wastes, March 1983, or APHA Standard Methods, 15 Edition. and EPA SW-846, 3rd Edition) (continued)

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Hexavalent Chromium, $Cr^{+6}$ | 200 ml for liquid sample<br>Approximately 50 grams for solid samples | Plastic or Glass | Cool, 4° C | 24 hours<br>Extracted within 28 days, analyzed within 7-days. of extraction. | Method 7196A<br>Method 7196 Colorimetric Method |

## E. GENERAL: MINERAL ANALYSIS/VOLATILES - DRINKING WATER or TITLE 22 CAL/DOHS

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Complete General Mineral Analysis | 1 - 2 liters | Plastic or Glass | Cool, 4° C, additional preservation depends on the analyte list | ----- | ----- |
| pH | 50 ml | Plastic or Glass | None | Immediate | EPA Method 150.1 pH Meter |
| Alkalinity | 50 - 100 ml | Plastic or Glass | Cool, 4° C | 14 days | EPA Method 310.1 Titrimetric Method |
| Calcium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 |
| Chloride | 50 - 100 ml | Plastic or Glass | Cool, 4° C | 28 days | EPA Method 325.3 Titrimetric Method; EPA 300.0 Ion Chromatography |
| Copper | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| MBAS | 500 ml | Plastic or Glass | Cool, 4° C | 48 hours | EPA Method 425.1 Colorimetric |
| Iron | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 |
| Magnesium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Manganese | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Sodium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 |
| Sulfate | 50 - 100 ml | Plastic or Glass | Cool, 4° C | 28 days | EPA 300.0 Ion Chromatography |
| Electrical Conductivity | 50 - 100 ml | Plastic or Glass | Cool, 4° C | 24 hrs. or filter | EPA Method 120.1 EC Water |
| Total Dissolved Solids | 100 ml | Plastic or Glass | Cool, 4° C | 7 days | EPA Method 160.1 Gravimetric |
| Total Hardness | 50 - 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | EPA Method 130.2 Titrimetric Standard Method 314-A Calculation |
| Zinc** | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Volatile Organics (VOAs) | 3 - 40 ml containers | Glass, Cap teflon-lined, 40 ml. | No headspace, HCl to pH<2, if residual chlorine then preserve with $Na_2SO_4$ or Ascorbic Acid Wash. State - No headspace and HCl to pH <2 only | 14 days | EPA Method 524.2 |

**** Each metal can also be analyzed by EPA 200.7 or EPA 6010A.**

## F. INORGANIC ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Arsenic | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Barium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Cadmium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Chromium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Lead | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP/MS EPA 200.8 |
| Mercury | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 28 days | EPA 245.1 or 245.2 Cold Vapor or ICP/MS EPA 200.8 |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Selenium | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP/MS EPA 200.8 |
| Silver | 100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | ICP EPA 200.7 or ICP/MS EPA 200.8 |
| Nitrate-Nitrogen | 50 ml | Plastic or Glass | Cool, 4° C add $H_2SO_4$ to pH <2 | 14 days | EPA 352.1, Brucine Sulfate |
| | | | Cool, 4° C | 48 hours | EPA 353.3, Cadmium Reduction |
| Fluoride | 300 ml | Plastic or Glass | None | 28 days | EPA 300.0 Ion Chromatography |

**** Each metal can also be analyzed by EPA 200.7 or EPA 200.8.**

## G. GENERAL PHYSICAL ANALYSIS: DRINKING WATER or TITLE 22 - CAL/DOHS

| PARAMETER | VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Color | 50 ml | Plastic or Glass | Cool, 4° C | 48 hours | EPA Method 110.2 |
| Odor | 200 ml | Plastic or Glass | Cool, 4° C | 48 hours | EPA Method 180.1, Threshold Odor |
| Turbidity | 100 ml | Plastic or Glass | Cool, 4° C | 48 hours | EPA Method 180.1, Nephelometric |

## H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS

| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Acidity | 100 ml | Plastic or Glass | Cool, ≤6° C | 14 days | EPA 305.1, Titrimetric |
| Alkalinity | 50-100 ml | Plastic or Glass | Cool, ≤6° C | 14 days | EPA 310.1 ,Titrimetric |
| Ammonia Nitrogen | 100 ml | Plastic or Glass | Cool, ≤6° C $H_2SO_4$ to pH <2 | 28 days | EPA 350.1, Colorimetric |
| BOD | 1 liter | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 405.1 |
| Boron | 100 ml | Plastic | $HNO_3$ to pH<2 | 6 months | ICP EPA 200.7 |
| Chloride | 100 ml | Plastic or Glass | None | 28 days | EPA Method 325.3, Mercuric Nitrate or EPA 300.0, Ion Chromatography |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| COD | 20 ml | Plastic or Glass | Cool, ≤6° C<br>$H_2SO_4$ to pH <2 | 28 days | EPA Methods 410.4, Colorimetric |
| Coliform<br>Fecal Coliform | 100 ml | Sterilized Plastic Bottles | Cool, 10° C<br>$Na_2S_2O_3$ preserved for presence of free chlorine | 6 hours or 30 hours depending on the test requested | Standard Method 909A or 909C |
| Color | 50 ml | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 110.2 or 110.3 |
| Cyanide | 500 ml | Plastic or Glass | Cool, ≤6° C<br>NaOH to pH >12 | 14 days | EPA 335.3, Colorimetric |
| Electrical Conductivity | 50 - 100 ml | Plastic or Glass | Cool, ≤6° C | 28 days | EPA 120.1, EC Meter |
| Fluorides | 300 ml | Plastic or Glass | None | 28 days | EPA 340.2, Ion Specific Electrode or EPA 300.0, Ion Chromatography |
| MBAS | 500 ml | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 425.1, Colorimetric |
| Nitrate Nitrogen | 100 ml | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 300.0, Ion Chromatography |
| Nitrate + Nitrite Nitrogen | 100 ml | Plastic or Glass | Cool, ≤6° C<br>Add $H_2SO_4$ to pH <2 | 28 days | EPA 353.3, Cadmium Reduction |
| Nitrite Nitrogen | 50 ml | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 354.1, Spectrophotometric or EPA 300.0, Ion Chromatography |
| Odor | 200 ml | Plastic or Glass | Cool, ≤6° C | 24 hours | EPA 140.1 |
| Oil and Grease | 1 liter | Glass | Cool, ≤6° C<br>Add HCl or $H_2SO_4$ to pH <2 | 28 days | EPA 1664 |
| Orthophosphate | 50 ml | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 365.2 |
| pH | 50 ml | Plastic or Glass | None | Analyze within 15 minutes | EPA Method 150.1, pH Meter |
| Phenolics | 500 ml | Glass | Cool, ≤6° C<br>Add $H_2SO_4$ to pH <2 | 28 days | EPA 420.1, 4-AAP |
| Radioactivity | 1-18 liters | Plastic or Glass<br>Glass only for Tritium | $HNO_3$ to pH <2 | 6 months | Standard Method 701 |
| Silica | 50 ml | Plastic | Cool, ≤6° C | 28 days | ICP EPA 200.7 |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Sulfates | 50 ml - 100 ml | Plastic or Glass | Cool, ≤6° C | 28 days | EPA 375.4, Turbidimetric or EPA 300.0, Ion Chromatography |
| Sulfides | 500 ml | Plastic or Glass | NaOH to pH >9<br>2 ml Zinc Acetate<br>Cool, ≤6° C | 7 days | EPA 376.1 ,Titrimetric |
| Sulfites | 50 ml | Plastic or Glass | None | Analyze within 15 minutes | EPA 377.1 ,Titrimetric |
| TOC | 25 ml | Plastic or Glass | Cool, ≤6° C<br>$H_2SO_4$ or $H_3PO_4$ to pH <2 | 28 days | EPA 415.1 or 9060, TOC Analyzer |
| Total Dissolved Solids | 100 ml | Plastic or Glass | Cool, ≤6° C | 7 days | EPA 160.1, Gravimetric |
| Total Hardness | 50-100 ml | Plastic or Glass | $HNO_3$ to pH <2 | 6 months | EPA 130.2, Titrimetric<br>Standard Method 314-A Calculation |
| Total Kjeldahl Nitrogen | 1 liter | Plastic or Glass | Cool, ≤6° C<br>Add $H_2SO_4$ to pH <2 | 28 days | EPA 351.4, Ion Specific Electrode |
| Total Organic Nitrogen TON-TKN-$NH_3$-N | 1 liter | Plastic or Glass | Cool, ≤6° C<br>Add $H_2SO_4$ to pH <2 | 28 days | EPA 351.4 and EPA 350.3 |
| Total Petroleum Hydrocarbons Scan, by GC | 400 ml to 1 liter<br>Approximately 50 grams for a solid sample | Glass | Cool, 4° C | 7 day if unpreserved<br>14 days if preserved but this may vary between states so regulations must be consulted<br>14 days | NWTPH-HCID by GC/FID or Modified Method 8015, GC |
| Total Petroleum Hydrocarbons as Gas, by GC | 3 - 40 ml containers<br>Approximately 20 grams for a solid sample | Glass | Cool, 4° C, Methanol or HCl preservation may be a requirement of some states so state regulations must be consulted<br>Cool, 4° C | 7 day if unpreserved<br>14 days if preserved but this may vary between states so regulations must be consulted<br>14 days | NWTPH-G by purge and trap GC/FID or Modified Method 8015, GC/FID AK101 |
| Total Petroleum Hydrocarbons as Diesel or oil, by GC | 1 liter<br>Approximately 50 grams for a solid sample | Glass | Cool, 4° C, Methanol or HCl preservation may be a requirement of some states so state regulations must be consulted<br>Cool, 4° C | 7 day if unpreserved<br>14 days if preserved but this may vary between states so regulations must be consulted<br>14 days | NWTPH-D by GC/FID or Modified Method 8015, GC/FID AK102 & AK103 |

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| PARAMETER | MINIMUM VOLUME REQUIRED | CONTAINER | PRESERVATION | MAXIMUM HOLDING TIME | METHOD FOR ANALYSIS |
|---|---|---|---|---|---|
| Total Phosphate | 50 ml | Plastic or Glass | Cool, ≤6° C<br>Add $H_2SO_4$ to pH <2 | 28 days | EPA 365.2, Colorimetric |
| Total Residue | 100 ml | Plastic or Glass | Cool, ≤6° C | 7 days | EPA 160.3 , Gravimetric |
| Total Settleable Solids | 1 liter | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 160.5, Imhoff Cone |
| Total Suspended Solids | 100 ml | Plastic or Glass | Cool, ≤6° C | 7 days | EPA 160.2, Gravimetric |
| Total Volatile Solids | 100 ml | Plastic or Glass | Cool, ≤6° C | 7 days | EPA 160.4, Gravimetric |
| TOX | 500 ml | Amber Glass, Teflon Septum | Cool, 4° C<br>Add $H_2SO_4$ to pH <2 | 7 days | EPA 450.1 or EPA 9020, TOX Analyzer |
| Turbidity | 100 ml | Plastic or Glass | Cool, ≤6° C | 48 hours | EPA 180.1 |

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# APPENDIX 8

## Checklist For Analyses With Short Holding Times

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## CHECKLIST FOR ANALYSES WITH SHORT HOLDING TIMES

To be completed prior to release of samples to the laboratory

_____ Correct test codes are entered

_____ Dates and times received and collected are correct

_____ Matrices are correct

_____ Sample I.D.s are correct

I certify that all of the above have been checked and were found accurately entered in the LIMS.

_____ _____
(Signature) (Date)

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4201

Title: **Package Deliverables for all Reporting Levels**

Revision history:

| Number | Date |
|---|---|
| 000 | 1/31/96 |

Written by: Tom Marino Date: 1-31-96

Reviewed by: Charlene Nix Date: 1-31-96

Reviewed by: Harry Romberg, QC Officer Date: 1-31-96

Approved by: Karen Kotz, Laboratory Director Date: 1/31/96

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## 1. Introduction and Scope

1.1 This SOP descibes the contents of the different levels of reports at Laucks Testing Laboratories, Inc.

1.2 Complete CLP-type data package (level IV):

A complete data package (level IV) submitted to a client consists of a cover page, a narrative, chain-of-custody copies, an index, and a separate section for each analytical fraction containing all forms and raw data. The entire package is paginated sequentially beginning with #1.

1.3 Forms only data package (level III):

A "forms only" data package (level III) submitted to a client contains all of the above with the exception of the raw data.

1.4 Database Report (level II):

A database report (level II) contains forms generated from the database and includes many of the forms in a level III or IV package. The contents are indicated in Appendix I and are not described further.

1.5 Paper Job Report (level I):

A "paper job" report is created for special chemistry and food chemistry and usually contains a format specified by the client or results only. The contents are indicated in Appendix I and are not described further.

## 2. Description of Contents of Level III and IV packages

Detailed below are the elements that may be included in a level III or level IV package. Appendix I contains tables that signify whether an element is incorporated into a final report. Client specific requests may dictate that some elements may be added or deleted and these are documented during project initiation in the LIMs system.

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2.1 Cover Page

The package cover page will contain the laboratory name, client name, work order numbers, SDG number and the date.

2.2 Narrative

2.2.1 Sample receipt and Identification:

This section lists all client sample names, the corresponding laboratory sample names and the analyses requested for each sample. (The analyses are generally abbreviated to three letters.)

2.2.2 Analytical request key:

This section defines the abbreviations listed in the above section.

2.2.3 Sample Identification on Forms:

This sections is used to explain any abbreviations to client sample names on any of the forms (occasionally forms software does not accommodate lengthy client sample I.D.s).

2.2.4 General remarks on organic analysis:

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested organic fractions.

2.2.5 Specific remarks on organic analysis:

These are comments written for each organic analytical fraction describing any anomalies, deviations from the specified method, dillutions, holding time violations, corrective actions etc. These comments are written by the respective analysts.

2.2.6 General remarks on inorganic analysis

These are stock comments contained in the narrative template describing general analysis conditions for each of the requested inorganics fractions.

2.2.7 Specific Remarks on inorganic analysis:

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These are comments written for each inorganic analytical fraction describing any anomalies,deviations from the specified method, dilution's, holding time violations,corrective actions etc. These comments are written by the respective analysts.

2.2.8 Release of data:

This page is signed by both the respective project manager and the technical director. also contained on this page is information on who to contact regarding specific questions as well as the laboratory telephone and fax numbers

2.3 Chain-of-Custody Copies:

This section contains the chains of custody received with the samples as well the laboratory receipt and temperature logs.

2.4 Index:
The index should list all data fractions and sub-fractions with the corresponding page numbers.

2.5 Organic fractions

Level IV Organic data packages are subdivided into five sections: QC Summary, Sample Data, Standards Data, Raw QC Data, and Bench Sheets.

Level III (forms only) organic data packages contain only the **forms** from these sections. No bench sheets or raw data are provided.

Only Volatiles, Semi-volatiles and Pesticides/PCBs have official CLP form numbers and protocol. Every effort is made to ensure that the same information appears on forms for all other fractions.

NOTE: the form numbers that appear below are seen only on forms for Volatiles, Semi-Volatiles and Pesticides/PCBs. Forms for all other fractions contain the same information but no actual form numbers.

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2.5.1 QC Summary:

The QC Summary contains the following forms:

a.) Form II: Surrogate recovery report
b.) Form III: MS/MSD Recovery report
c.) Blank spike report
d.) Form IV: Method blank summary
e.) Form V: Tuning and Mass Calibration Standard
f.) Form VIII: Internal standards Area Summary

2.5.2 Sample Data:

Sample data contains the following forms and data

a.) Form I (analysis data sheet) including TICs
b.) Raw Data

2.5.3 Standards Data:

The standards data below are divided into two formats: Volatile/Semi-Volatile and Pesticide/PCB. Every effort is made to ensure that forms for all other fractions adhere closely to whichever of the two formats is most applicable.

2.5.3.1 Volatile/Semi-Volatile

a.) Form VI and Initial Calibration Data
b.) Form VII and Continuing Calibration Data

2.5.3.2 Pesticide/PCB

a.) Form VIII: Pesticide Analytical Sequence
b.) Form IX: Pesticide/PCB Standards Summary
c.) Form X: Pesticide/PCB Identification (positive results)
d.) Pesticide standard chromatograms and data system printouts for Evaluation of standard mix A,B, and C
e.) Pesticide standard chromatograms and data system printouts for individual standard mix A, and B
f.) Pesticide Standard Chromatograms and data system printouts for all multi-response pesticides/PCBs and quantitation standards
g.) A copy of the computer reproduction or strip chart recorder output covering the 100 fold range

2.5.4 Raw QC Data:

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The Raw QC Data below are divided into three formats: Volatile, Semi-Volatile, and all other fractions.

2.5.4.1 Volatile
- a.) BFB
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.2 Semi-Volatile
- a.) DFTPP
  - 1.) Bar graph spectrum
  - 2.) Mass listing
  - 3.) RIC: Reconstructed Total Ion Chromatogram
- b.) Blank Data
  - 1.) Form I including TICs
  - 2.) Raw data
- c.) Matrix Spike Data
  - 1.) Form I
  - 2.) Raw data
- d.) Matrix Spike Duplicate Data
  - 1.) Form I
  - 2.) Raw data

2.5.4.3 All other fractions
- a.) Blank Data
  - 1.) Form I
  - 2.) Raw data

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b.) Matrix Spike Data
  1.) Form I
  2.) Raw data
c.) Matrix Spike Duplicate Data
  1.) Form I
  2.) Raw data

2.5.5 Bench Sheets:

The bench sheets section contains any miscellaneous paper work such as log book pages, in-house tracking sheets, in-house chains of custody, extraction bench sheets etc.

2.6 Inorganics Fractions:

There are two kinds of inorganics fractions: Metals and Miscellaneous Inorganics (conventionals).

2.6.1 Metals

2.6.1.1 Cover Page:

This page lists all samples analyzed and is signed and dated by the analyst

2.6.1.2 Inorganics Analysis Data Sheet
a.) forms I

2.6.1.3 Quality Control Data
a.) Form II (part 1): Initial and Continuing Calibration Verification
b.) Form II (part 2): CRDL Standard for AA and ICP
c.) Form III: blanks
d.) Form IV: ICP Interference Check Sample
e.) Form V (part 1): Spike Sample Recovery
f.) Form V (part 2): Post Digest Spike Sample Recovery
g.) Form VI: Duplicates
h.) Form VII: Laboratory Control Sample
i.) Form VIII: Standard Addition Results
j.) Form IX: ICP Serial Dilutions
k.) Form XIII: Preparation Log
l.) Form XIV: Analysis run Log

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2.6.1.4 Quarterly Verification of Instrument Parameters

a.) Form X: Instrument Detection Limits (quarterly)
b.) Form XI (part 1): ICP Inter element Correction Factors (annually)
c.) Form XI (part 2): ICP Inter element Correction Factors (annually)
d.) Form XII: ICP Linear Ranges (quarterly)

2.6.1.5 Raw Data

a.) ICP
b.) Graphite Furnace
c.) Mercury
d.) Cyanide

2.6.1.6 Digestion and Distillation Logs

a.) ICP
b.) Graphite Furnace
c.) Mercury
d.) Cyanide

2.6.2 Miscellaneous Inorganics

2.6.2.1 Cover Page

This page lists all samples analyzed and is signed and dated by the analyst.

2.6.2.2 Inorganics Analysis Data Sheet

2.6.2.3 Quality Control Data

a.) Preparation blanks database Report
b.) MS/MSD Report
c.) MS/Duplicate Database
d.) SRM Report

2.6.2.4 Raw Data

The Miscellaneous Inorganics raw data is divided into sections by analytes.

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Appendix I

## Laucks Testing Laboratories
## Organics
## Levels of Reporting

| | Level I | Level II | Level III | Level IV = CLP |
|---|---|---|---|---|
| Narrative | | | Y | |
| Chain-of-Custody | Y | Y | Y | Y |
| Method Reference | Y | Y | Y | Y |
| Results | Y | Y | Y | Y |
| Surrogate Rec. | | Y | Y | Y |
| Method Blank | | Y | Y | Y |
| Blank Spike | | Y | Y | Y |
| MS/MSD | | Y | Y | Y |
| Tune | | | Y | Y |
| Initial Calib. | | | Y | Y |
| Cont. Calib. | | | Y | Y |
| IS Area | | | | Y |
| Raw Data | | | Optional | Y |
| Chromatograms | | | Optional | Y |
| Bench Sheets | | | | Y |

Y indicates that this element is present in the hardcopy delivered to the client.

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## Laucks Testing Laboratories
## Metals
## Levels of Reporting

| | Level I | Level II | Level III | Level IV = CLP |
|---|---|---|---|---|
| Narrative | | | Y | Y |
| Chain-of-Custody | Y | Y | Y | Y |
| Method Reference | Y | Y | Y | Y |
| Results | Y | Y | Y | Y |
| Method Blank | | Y | Y | Y |
| SRM/LCS/BS | | Y | Y | Y |
| MS/Dupe | | Y | | Y |
| MS/MSD | | | Y | Y |
| ICV | | | Y | Y |
| ICB | | | Y | Y |
| CCV | | | Y | Y |
| CCB | | | Y | Y |
| CRA | | | Y | Y |
| CRI | | | Y | Y |
| Post Spike | | | Y | Y |
| GFAA MSA | | | Y | Y |
| ICP Ser. Dil'n. | | | Y | Y |
| IDLs | | | Y | Y |
| Interelement Corr. Factors | | | | Y |
| Linear Range | | | | Y |
| Prep Log | | | | Y |
| Run Log | | | | Y |
| Raw Data | | | | Y |
| Bench Sheets | | | | Y |

Y indicates that this element is present in the hardcopy delivered to the client.

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**Laucks Testing Laboratories**
**Wet Chemistry**
**Levels of Reporting**

| | Level I | Level II | Level III | Level IV = CLP |
|---|---|---|---|---|
| Narrative | | | Y | Y |
| Chain-of-Custody | Y | Y | Y | Y |
| Method Reference | Y | Y | Y | Y |
| Results | Y | Y | Y | Y |
| Method Blank | | Y | Y | Y |
| SRM/LCS/BS | | Y | Y | Y |
| MS/Dupe | | Y | | Y |
| MS/MSD | | | Y | |
| ICV | | | Y | Y |
| ICB | | | Y | Y |
| CCV | | | Y | Y |
| CCB | | | Y | Y |
| Raw Data | | | | Y |
| Bench Sheets | | | | Y |

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-7003

Title: **Inorganic Glass Cleaning Procedure**

Revision history:

| Number | Date |
|---|---|
| 1 | 01/05/88 |
| 2 | 04/09/97 |
| 3 | 06/30/03 |
| 4 | 6/30/05 |

Revised by: Bill Ambacher Date: 6/30/05
Bill Ambacher, Inorganics Manager

Reviewed by: Harry Romberg Date: 7/18/05
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 7-19-05
Kathy Kreps, Laboratory Director

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 The purpose of this SOP is to define the procedures used in the inorganics department for the cleaning of glassware. The objective is to define a specific method of cleaning that is adapted to both the substances that are to be removed, and the determination to be performed.

1.2 Definitions

1.2.1 DIW - Deionized water- Lab reagent water. This water should be free of virtually all analytes.

## 2. Equipment List

2.1 Standard Laboratory Glassware

2.1.1 Volumetric flasks
Beakers
Funnels
Separatory funnels
Kjeldahl flasks
Nessler tubes
Erlenmeyer flasks
Burets
BOD bottles
Distillation Apparatus

Other Assorted Glassware

## 3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 Several cleaning procedures or soaking procedures require the use of concentrated $HNO_3$ or $H_2SO_4$. Use appropriate safety precautions for acid use! Wear safety glasses, lab coat, and gloves.

3.1.2 Some oily samples or profile samples may require the use of acetone or chloroform to clean the glassware. Wear safety glasses, lab coat, and gloves. Dispose of solvent waste in appropriate solvent waste barrel.

3.1.3 Some profile-type samples may not clean up even with solvent washes, and therefore may need to be baked in the muffle furnace at 500 degrees C for 1-3 hours.

| |
|---|
| Note: Never muffle glassware at greater than 500°C or it may deform or melt. Never muffle volumetric glassware or it may deform enough to change volume. |

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3.1.4 CAUTION: Be sure to evaporate any residual solvent from glassware before putting in muffle furnace.

3.1.5 Do not put soft glass (non-pyrex, kimax, etc.) in a muffle furnace or it will shatter. Take appropriate precautions with hot materials.

## 4. Operation procedures

4.1.1 All glassware must be scrupulously cleaned. The analyst that performs each specific analysis is responsible for the proper cleaning of his or her own glassware. Glassware used in routine analysis is kept separate from the general use glassware. Specific cleaning procedures are listed by type of analysis.

4.2 ALKALINITY

4.2.1 Glassware: Erlenmeyer flasks, Buret

4.2.2 Cleaning Procedures - Rinse with DIW.

4.2.3 Air dry.

4.3 AUTO ANALYZER (refer to the applicable analytical SOP)

4.4 BOD

4.4.1 Glassware: BOD bottles, glass stoppers

4.4.2 Cleaning procedure - Wash with hot tap water and Alconox.

4.4.3 Rinse with hot tap water.

4.4.4 Rinse with DIW.

4.4.5 Air dry.

4.5 CYANIDE

4.5.1 Glassware: Volumetric flasks, Distillation apparatus

4.5.2 Cleaning procedure for Flasks – Rinse with hot water twice followed by DIW rinse twice.

4.5.3 Distillation apparatus - occasional $H_2SO_4$ and DIW wash, but generally DIW rinsed only.

4.6 HEXAVALENT CHROMIUM

4.6.1 Glassware: Nessler Tubes

4.6.2 Cleaning procedure - Wash with hot tap water.

4.6.3 Rinse well with DIW.

4.6.4 Air dry.

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4.7 FORMALDEHYDES

4.7.1 Glassware: Beakers, Erlenmeyer flasks, Volumetric flasks, Test tubes

4.7.2 Cleaning procedures - Rinse well with DIW only.

4.7.3 Never contaminate glassware with $HNO_3$.

4.8 HARDNESS

4.8.1 Glassware: Erlenmeyer flasks, Buret

4.8.2 Cleaning procedure - Rinse with DIW only.

4.9 KJELDAHL NITROGEN-LOW LEVELS (TKN AND AMMONIA)

4.9.1 Glassware: Kjeldahl distillation apparatus, Kjeldahl flasks, Erlenmeyer flasks or beakers

4.9.2 Cleaning procedure (must be done immediately before use to minimize airborne ammonia that may adhere to glassware) – Rinse two times with hot water.

4.9.3 Rinse twice with DIW.

4.10 OIL AND GREASE – Method 1664

4.10.1 Glassware: Beakers, Funnels, Soxhlet digestion apparatus

4.10.2 Cleaning procedure for funnels - hexane rinse 3-4 times.

4.10.3 Cleaning procedure for soxhlet apparatus – Rinse with DIW, then acetone

4.10.4 Cleaning procedure for Beakers- hot tap water and Alconox, rinse well with hot tap water, then DIW and dry in oven.

4.11 METALS (INCLUDING MERCURY)

4.11.1 Glassware: Volumetric flasks, Beakers, Erlenmeyer flasks, or watchglasses

4.11.2 Cleaning procedure - Rinse well with DIW.

4.11.3 Fill with 5% HNO3 and store covered until next use.

4.11.4 OPTIONAL:

4.11.5 If oily or difficult to clean, wash with acetone, scrub with hot tap water and Alconox, and rinse well with DIW. Care must be taken to ensure that any residual Alconox is rinsed off the glassware, or it may contribute to blank contamination.

4.11.6 Concentrated acid wash and DIW rinsed.

4.11.7 Bake in muffle furnace at 500°C for 1 hour and rinse well with DIW.

4.11.8 NOTE: Some metals glassware should not be cleaned with Alconox at all.

4.11.9 Soaking procedure - Acid soak all metals glassware in $HNO_3$ and DIW.

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4.11.9.1 Fill glassware with DIW and add 4-8 mls $HNO_3$. Cover and store until next use.

4.12 NITROCELLULOSE

4.12.1 Wash with Hot Water.

4.12.2 Rinse with acetone

4.13 PHENOL

4.13.1 Glassware: Distillation flasks, condensers, volumetric flasks and separatory funnels.

4.13.2 Cleaning procedure for distillation flasks - pre-distill with dilute $H_2SO_4$. DIW rinse.

4.13.3 Cleaning procedure for volumetric flasks and separatory funnels. Rinse with DIW 2 times, acetone 2 times, then chloroform.

4.14 PHOSPHATE

4.14.1 Glassware: Erlenmeyer flasks.

4.14.2 Acid wash with HCl.

4.14.3 Rinse with DIW.

4.14.4 Store in 1% HCl

Note: Detergents of all kinds must be avoided as most contain anywhere from a trace amount to high levels of phosphate.

4.15 TOC (SOILS)

4.15.1 TOC combustion boats

4.15.2 Cleaning procedure - Brush out last sample.

4.15.3 Incinerate boat in muffle furnace at 950 degrees C.

4.16 TOX

4.16.1 Glassware: Volumetric flasks, Misc. TOX glass parts

4.16.2 Wash with hot tap water.

4.16.3 Rinse well with DIW.

4.16.4 Air Dry

4.17 TOTAL SOLIDS

4.17.1 Glassware: Beakers (do not use scratched beakers)

4.17.2 Dissolve residue with HCl

4.17.3 Rinse with DI water and scrub with brush (avoid scratching beakers)

4.17.4 Place in oven to dry until needed

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## 5. References

5.1.1 See the applicable analytical or preparation SOP for specific cleaning issues and references.

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-7105

Title: Method Protocols for the Analysis of Metals by ICP Using SW 846 6010B

Revision history:

| Number | Date |
|---|---|
| 0 | 1/29/98 |
| 1 | 03/24/00 |
| 2 | 12/21/01 |
| 3 | 10/17/02 |
| 4 | 1/8/07 |

Revised by: Bill Ambacher, Chemist Manager Date: 1/8/07

Reviewed by: Harry Romberg, Quality Assurance Officer Date: 1/9/07

Approved by: Hugh Prentice, Laboratory Director Date: 1/9/07

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## 1. Introduction and Scope

1.1 Scope

1.1.1 This method is to be used as a supplement to the instrumental SOP, LTL-7106, in order to follow the method requirements of SW846 6010B for ICP analysis. Operating parameters are to be followed in the individual instrument SOP.

1.1.2 The analyst should become familiar with SW846 protocols prior to using this SOP.

1.2 Personnel Qualifications

1.2.1 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are to be collected in either glass or plastic containers. Water samples are to be preserved to a pH < 2 using nitric acid. A one-liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation but need to be stored at 4±2° C. At least 200 grams of sample should be collected. The holding time for ICP metals is 6 months. See Appendix III for a Sample Handling and Preservation Table.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

**ICV** - Initial Calibration verification - This is a standard run immediately following the initial calibration. The ICV is made from an independent source. Agreement within 10% and a relative standard deviation less than 5% RSD from replicate (minimum of two) integrations is required.

**ICB** - Initial calibration blank - An instrument blank is made up in the same way as calibration standards, without target analytes.

**CCV** - Continuing calibration verification - This is a standard analyzed after every 10 samples during the analysis sequence to determine whether the instrument or system has remained in calibration. Agreement within 10% and less than 5% RSD from replicate (minimum of two) integrations, is required.

**CCB** - Continuing Calibration Blank - This is a blank that is used to determine if there is carry-over between sample injections. A CCB immediately follows every CCV.

**CRDL** - Contract Required Detection Limit – A QC standard at a minimum level of detection, which must recover at ±20% of the true value. This limit represents the analyte concentrations below which data must be evaluated and qualified as estimated concentrations. The commercial CRDL solution may be used as a check standard, but must be fortified with As, B, Ba, Mo, Pb, and Se.

**DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

**IEC** - Interelement Correction Factors - IECs are experimentally-determined constants used by the calibration modeling software in estimating the extent of interference caused by IEC elements on the other analyzed elements. This list is to include the nine interferents listed in SW846 Method 6010B - Al, Ca, Cr, Cu, Fe, Mg, Mn, Tl, and V.

**ICSA** - Interference Check Solution A - This solution is prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors.

**ICSAB** - Interference Check Solution AB - This solution is prepared by spiking the ICSA with known quantities of analyte. Adequate recovery of the analytes within this interfering matrix indicates proper application of the correction factors.

**IDL** - Instrument detection limit - The lowest concentration of a target analyte that is detectable. The IDL is three times the average standard deviation of seven replicates at 2 to 5 times the estimated IDL over three non-consecutive days. Used as a starting point for selecting MDL study spiking levels. IDLs should be determined quarterly.

**Method Blank Sample/Preparation Blank Sample** - A sample containing only reagent and representative matrix (DIW or blank solid material, as appropriate). This sample is to be carried through the entire preparation procedure and analyzed as a regular sample. The level of analytes in this sample must not exceed one-half the concentration requested as reporting limits for the project. Corrective action will be either qualification of data associated with that blank sample or repreparation/reanalysis of the associated samples.

**MS** - Matrix spike - The matrix spike sample is to be prepared, such that the amounts of analyte added are near the action levels of concern for the project, but above 20x the MDLs. Preparation of the matrix spike sample is described in the appropriate SOP.

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**MDL** - Method detection limit - The lowest concentration which will yield a positive result that is greater than zero at a known level of confidence. The MDL is preparation specific and empirically determined by Laucks.

**LCS** - Laboratory Control Sample - This is a material of approximately the same matrix as the samples, containing a known and usually certified amount of target analyte and which is prepared and analyzed in the same manner as a typical sample. This sample is used to demonstrate that the analytical system is in control. It may be considered to be a blank spike for most inorganic analyses and is preferred over artificially spiking blank materials.

**Post digestion spike** - The sample selected for QC analysis will be post-spiked and analyzed in the event that the matrix spike sample exceeds recovery criteria. The final spike level should be identical to the predigestion spike, near the action levels of concern for the project but above 20x the MDL.

**QC period** - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank analysis.

**RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values to each other.

**Sequence** - A set of sample digests and standard solutions introduced into an instrument in a chronologically continuous group.

## 2. Equipment List and Standards

2.1 Instrumentation

2.1.1 Thermo Jarrell Ash ICAP 61E Trace Analyzer or equivalent.

2.2 Computer and software

2.2.1 Pentium 75 computer (or faster), running Thermospec software version 6.2 or later.

2.2.2 iLIMS (Laucks laboratory information management system) or Metals Analytical Review and Reporting System (MARRS), version 4.0.1 or equivalent for CLP data reporting.

2.3 Standards

2.3.1 SW846 Method 6010B requires the use of one standard and a blank for calibration. Laucks uses five standards, which contain various elements. Only K, Ca, Mg, Na, Fe,

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and Al are present in all five standards. All other elements are present in one standard. Laucks confirms the low end of the calibration with a check standard (CRDL) that must be recovered within ± 20% of true value.

## 3. Safety precautions and Waste Disposal

3.1 Safety

3.1.1 See the instrument SOP (LTL-7106) for detailed safety precautions

3.1.2 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.3 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.4 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.5 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.6 Caution should be used when handling acidic digestates.

3.2 Waste Disposal

3.2.1 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal, LTL-2001. All acidified samples and standards should be neutralized prior to sewer disposal.

## 4. Calibration and Quality Control

4.1 Method Detection Limit Study

4.1.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on performing MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration approximately 3 to 5 times the estimated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. See Appendix VII for estimated MDLs. Current MDL values are found in the iLIMS MDL database and associated raw data are stored in the metals area.

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4.2 Linear range study

4.2.1 The upper limit of the linear dynamic range must be established by running a multi-point calibration using 4 standards and then running an upper range standard. The upper range limit should be an observed signal with no more than a 10% variation from the true value of the upper range standard. All samples with elements exceeding the level of the upper range standard must be diluted.

4.2.1.1 The linear range should be checked every six months by analyzing a standard at the determined linear range. The standard must fall within 10% of the true value. If the standard does not fall within 10%, a new linear range must be determined as described in 4.2.1.

4.3 Initial Calibration and Preliminary Calibration Check Standards

4.3.1 Calibrate using a blank and five standards (not all elements are in all standards). The calibration curve must be verified by analyzing an Initial Calibration Standard (ICV) and obtaining agreement within ±10% of the expected concentration with an RSD < 5% for 2 replicate integrations.

4.3.2 One check standard at the CRDL is to be analyzed, and the recovery criteria are ±20% of true value.

4.3.3 Criteria and Corrective Action:

4.3.3.1 Calibration – The standard curve is validated by evaluating the ICV and the subsequent CCVs. If the corresponding control limits for the ICV and CCV are exceeded, then the sample analysis must be discontinued for the affected elements, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV/CCV must be reanalyzed (for the affected elements).

4.3.3.2 Linear range standard – If this standard is not included in the analysis, then any data acquired with concentration values above the high standard of the curve, but within the linear range of the instrument must be qualified as estimated quantities. Preferably, the samples would be diluted and reanalyzed.

4.3.3.3 CRDL – If the stated criterion cannot be met, then samples with concentrations between the CRDL and the calibration standard can not be reported.

4.4 Initial Calibration Blank

4.4.1 After the analysis of the high standard, an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should not exceed ½ of the reporting limit.

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4.4.2 Corrective Action:

4.4.2.1 The source of contamination must be identified and corrected before proceeding with the analysis.

4.5 Continuing Calibration Verification (CCV) and Blank (CCB)

4.5.1 A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank solution (CCB) is analyzed. In addition, this standard and blank must be the last samples analyzed in the run.

4.5.1.1 The CCV must fall within ± 10% of the true value and the RSD must be less than 5% for replicate integrations.

4.5.1.2 The levels of target analytes in the CCB should not exceed ½ of the reporting limit.

4.5.2 Corrective action:

4.5.2.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze for the affected elements. No sample results may be reported that are not bracketed by a successful calibration and CCV/CCB which are in control or by preceding and following CCV/CCBs which are within limits.

4.5.2.2 If the CCB analyte levels exceed ½ of the reporting limit, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If sample concentrations are significantly higher than the level in the CCB (usually 10-20x), then the results may be reported with a narrative comment.

4.6 Interference Check Solutions A (ICSA) and AB (ICSAB)

4.6.1 ICSA: At the beginning of each run, an interference check solution A is analyzed. This solution contains interfering elements only. All other elements are not present in the solution. All elements not present should show a recovery of within ± the CRDL.

4.6.2 Corrective Action:

4.6.2.1 If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning another run.

4.6.3 ICSAB: At the beginning of each analytical sequence, an ICSAB must be analyzed. Analytes must recover between 80-120% of true value.

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4.6.4 Corrective Action:

4.6.4.1 If the analytes do not recover within the specified control limits, then the system is out of control. The problem needs to be identified and corrected prior to beginning analysis.

4.7 Method Blanks

4.7.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, which ever is more frequent. Any analyte response above the IDL is reported. For a method blank to be acceptable for use with the accompanying samples, the concentration of the blank of any analyte of concern should not be higher than the highest of either:

(1) One-half of the reporting limit,
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

4.7.2 Corrective Action:

4.7.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example, if an analyte was detected in the blank but not in any of the associated samples, then the sample group may not require re-analysis. In any case, if re-preparation and re-analysis is not being undertaken, the analyst must first discuss the issue with the Quality Control Officer. It is the laboratory's responsibility to ensure that method interference caused by contaminants in acids, solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the analytical run be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source, so it can be eliminated. In all cases where blank contamination exceeds the control limit, a narrative comment must be made which documents the corrective actions taken.

4.8 Laboratory Control Sample/Blank Spike

4.8.1 A LCS (for water digestions, and soils, if requested) or a blank spike is carried through the entire digestion procedure and is performed with each digestion batch. At a minimum, the LCS (water) or the Blank Spike control limits, are 80% to 120% of true value.

4.8.2 If requested, a matrix specific soil LCSS is digested with samples. The manufacturer supplies LCSS (soil) control limits. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

4.8.3 Corrective Action

4.8.3.1 If the LCS/BS is not within the required control limits but the matrix spike is in control, corrective action may not be required unless otherwise specified. If both LCS/BS and matrix spikes exceed limits, particularly if the recoveries are low or there are reportable quantities of the affected target analytes, then a redigestion will occur for the affected analytes. Other extenuating circumstances may exist that allow for exceptions without redigestion and reanalysis but they must first be approved by QA.

4.9 Matrix Spike

4.9.1 Unless specific samples are identified for use as matrix spike samples, a sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to preparation. Spike samples will be identified on the SDG Routing Forms as required. The analyst should attempt to avoid selecting samples that are identified by the client as blanks.

4.9.2 The matrix spike sample is to be prepared such that the amounts of analyte added are near the action levels of concern for the project, but above 20x the MDLs. Preparation of the matrix spike sample is described in LTL-7106.

4.9.3 As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$recovery,\% = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spike added, the amount of spiking material actually added calculated on the sample basis.

4.9.4 For ICP, control limits for spike recoveries will be 75-125% unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance.

4.9.5 Corrective Action:

4.9.5.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the LCS and/or blank spike analysis.

4.9.5.2 In some cases, a Post Digestion Spike is required when matrix interference is suspected. The sample selected for QC analysis will be post-spiked and analyzed in the event that the matrix spike sample exceeds recovery criteria. The final spike level should be identical to the predigestion spike. The same 75-125% recovery criteria exist for this spike as for the matrix spike. If the post-digestion spike exceeds these limits, then a matrix effect is suspected.

4.9.5.3 When a matrix spike and a post spike are recovered outside the control limits of 75%-125% recovery, then the analyst will reanalyze the matrix spike and post spike. Dilution of the matrix spike and post spike may also be needed to demonstrate recovery.

4.9.5.4 If the analyst cannot use dilutions to recover matrix and post spikes within the control limits of 75%-125% recovery, then a non-routine option is the Method of Standard Additions (MSA) described in SW846 method 7000. This technique involves adding known amounts of standard to one or more processed sample aliquots. The sample concentration is then calculated according to SW846 method 7000.

*Note: Since Laucks routinely uses an internal standard during analysis to prevent matrix affects on results, the Method of Standard Additions is only mentioned as a non-routine option.*

4.9.5.5 In all cases, a narrative explanation of the condition is required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event.

4.10 Matrix Spike Duplicate/Sample Duplicate

4.10.1 Method QC consists of MS/MSD. A duplicate may be performed instead of a MSD. Other types of QC can be performed at the client's request.

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4.10.2 Criteria

4.10.2.1 At least one matrix spike duplicate sample per 20 samples per matrix is required when matrix spikes are being performed. RPD values are calculated in a manner similar to MS/MSD RPDs:

$$RPD = \frac{|SS - SSD|}{(SS + SSD)/2} * 100$$

where:
SS = concentration in spiked sample
SSD = concentration in matrix spiked duplicate sample

4.10.2.2 For sample concentrations greater than five times the CRDL, control limits for the RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than five times the CRDL, control limits for the difference will be ± the reporting limit. In-house control limits are based on historical performance.

4.10.3 Corrective Action:

4.10.3.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples; no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Control Officer. In a validatable package, data associated with an out-of-control sample duplicate will be flagged with an "*".

4.11 Serial Dilution

4.11.1 A five-fold dilution is performed on the QC sample in each analytical batch. The difference between the initial value and the serial dilution should agree within 10%. In a validatable package, if the difference is greater than 10% on analytes that exhibit a level 50 times greater than the MDL, then results for those analytes will be flagged with an "E".

4.11.2 Corrective Action:

4.11.2.1 No corrective action is necessary other than appropriately flagging the data.

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## 5. Operation procedures

5.1 Instrumental Conditions

5.1.1 See instrument SOP for operating procedures, LTL-7106.

5.2 Analysis Sequence

5.2.1 See Appendix V for Analytical Sequence

5.3 Analytical Operation

5.3.1 See instrument SOP for operating procedures and standard concentrations, LTL-7106.

## 6. Troubleshooting

6.1 Troubleshooting

6.1.1 Analysts perform the majority of their own maintenance/troubleshooting on these instruments. They have available to them instrument manufacturer manuals (see references below), instrument maintenance manuals (which include descriptions of previous problems and how they were solved), and can call instrument manufacturer technical support. Instrument software can also be a source for maintenance/troubleshooting.

## 7. Reports

7.1 Data Packet Organization

7.1.1 See the metals validation SOP, LTL-7001, for a check list detailing data packet organization

7.1.2 If requested, all analysis performed under SW 846 guidelines can be reported via CLP SOW ILM04.1 forms. These forms provide all relevant QC information.

7.1.3 Data packages will be produced via iLIMS. Analyte levels that are less than the IDL or MDL will be reported as the SDL followed by a "U". Analyte levels that fall between the IDL or MDL and the reporting limit will be flagged with a "B". Analyte levels greater than or equal to the reporting limit PQL will be reported without a flag.

7.2 Flagging

| CODE | Definition |
|---|---|
| U | The analyte of interest was not detected, to the limit of detection indicated. |

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| | | |
|---|---|---|
| B | The analyte of interest was detected between the MDL and the reporting limit. | ❁ |
| N | The spike recovery exceeded the control limits. | ❁ |
| * | The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit. | ❁ |
| E | The Serial Dilution did not agree within 10%. | ❁ |
| S | The analyte concentration was determined by MSA. | ❁ |

❁ ◆ Used in all reports.
❁ Used in data validatable packages.
Other flags may be required but are specified in project specific documents.

7.3 Control Chart(s)

7.3.1 The recovery values for a selection of target metals in the LCS/Blank Spike are plotted on control charts. Other analytes may be added at the discretion of QA without immediate revision of this SOP.

7.4 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6010B, Revision 2, December 1996

Department Of Defense Quality Systems Manual for Environmental Laboratories, Version 3, January 2006

Air Force Center for Environmental Excellence (AFCEE) Quality Assurance Project Plan, Version 4.0.02, May 2006

ICAP 61E Trace Analyzer Operator's Manual, ThermoJarrell Ash Corp., 2001

2003 NELAC Standard, National Environmental Laboratory Accreditation Conference, June 5, 2003.

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**Appendix I**

**QC Summary Table**

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## Laucks Testing Laboratories

### Method 6010B QA Requirements and Corrective Actions

(See Appendix II for Agency Specific Criteria)

| QA Element | Method Criterion | Laucks Criterion | Frequency | Corrective Action | Documentation |
|---|---|---|---|---|---|
| Initial Calibration | One standard and a Blank | One standard and a Blank | Beginning of each run. | | In raw data. |
| Initial Calibration Verification (ICV) | ±10% of nominal value and RSD < 5% for replicate integrations. Made from an independent source. | ±10% of nominal value and RSD < 5% for replicate integrations. Made from an independent source. | Beginning of each run immediately following ICAL. | Recalibration required. | Form 2A or in raw data. |
| Continuing Calibration Verification (CCV) | ±10% and RSD < 5% for replicate integrations. | ±10% and RSD < 5% for replicate integrations. | Every 10 samples. Mid-range | Recalibrate and rerun affected samples. | Form 2A or in raw data. |
| Instrument Blank (ICB/CCB) | ±3 times the IDL. | < ½ reporting limit | Every 10 samples. | Recalibrate and rerun affected samples. | Form 3 or in raw data. |
| Method Blank | None | < ½ the reporting limit or <5% of the regulatory limit or <5% of the sample conc. (whichever is highest). | One/batch | Flag data per QCO or redigest samples | Form 3, in raw data, or database report. |
| Laboratory Control Sample/Blank Spike | None | LCSW/Blank Spike: 80%-120% LCSS: Manufacturer Specs. | One/batch | Redigest samples | Form 7, in raw data, or database report |

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## Laucks Testing Laboratories

### Method 6010B QA Requirements and Corrective Actions

(See Appendix II for Agency Specific Criteria)

| QA Element | Method Criterion | Laucks Criterion | Frequency | Corrective Action | Documentation |
|---|---|---|---|---|---|
| Matrix Spike Recovery | 75-125% or within documented historical limits | 75-125% or current QC database criteria. | 5% or per batch | Consult QCO | Form 5, in raw data, or database report |
| Post Digestion Spike Recovery | 75-125% used for new or unusual sample matrices | 75-125%. Spike level either 2x RL or 2x native sample level, whichever is greater | Used when MS is out of control | None | Form 5B, in raw data, or database report |
| Duplicate % Difference | ± 20% if sample conc. >10x the IDL | ± 20% or current QC database criteria. ±RL if sample is <10x the IDL | 5% or per batch | Consult QCO | Form 6, in raw data, or database report |
| Serial Dilution | ± 10% difference used for new or unusual matrices | ± 10% difference if sample conc. > 50x the MDL | One/batch | Flag data with an "E". | Form 9 or in the raw data. |
| ICSA,AB | ±20% true value of analytes, or ± the CRDL. | ±20% true value of analytes, or ± the CRDL. | Beginning and end of run | Recalibrate and rerun affected samples | Form 4, or in raw data. |
| Standard Reference Material (SRM) Recovery | See QC control catalog | Control limits set by vendor | 5% or per batch | Redigest samples | Form 7, in raw data, or database report |
| CRDL check standard | No criteria | ±20% true value of analytes, or per client requirement | Beginning of the analysis immediately following ICV/ICB | Recalibrate and reanalyze | Form 2B, or in raw data. |

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**Appendix II**

**Agency Criteria**

| Study/QC | 6010B | | DOD-QSM (version 3, 2006) | | AFCEE (4.0.02) | | ILM 4.1 | | ILM 5.3 | | 200.7 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency |
| IDLs | --- | Initially | --- | 3 mo. | --- | --- | --- | 3 mo. | --- | --- | --- | --- |
| MDLs | --- | Initially | --- | 12 mo. | --- | 12 mo. | --- | --- | <1/2 CRQL | 12 mo. | --- | 12 mo. |
| Linear Range Study | --- | Initially | --- | --- | --- | --- | --- | Initially | --- | Initially | +/- 10% | Initially |
| Linear Range Check Std. | +\- 10% | 6 mo. | +/- 10% | 6 mo. | +/- 10% | 3 mo. | +/- 5% | 3 mo. | +/- 5% | 3 mo. | +/- 10% | 12 mo |
| IECs | --- | 6 mo. | --- | 6 mo. | --- | | --- | 12 mo. | --- | 3 mo. | --- | 12 mo. |
| ICV/CCV | +/- 10% | Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV) | +/- 10% | Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV) | +/- 10% | Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV) | +/- 10% | Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV) | +/- 10% | Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV) | ICV +/- 5%, CCV +/- 10% | Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV) |
| ICB/CCB/PB | No analytes detected >= 3x IDL (ICB/CCB). No criteria (PB) | Immed. after calib.(ICB), prior to & after every 10 samples (CCB), and at end of run (CCB). One per prep batch (PB) | No analytes detected > 2x MDL (ICB/CCB). No analytes detected > 1/2 RL (PB) | Immed. after calib.(ICB), prior to & after every 10 samples (CCB), and at end of run (CCB). One per prep batch (PB) | No analytes detected >= 2x MDL | Immed. after calib.(ICB), after every 10 samples (CCB), and end of run (CCB) | <= CRDL | Immed. after calib.(ICB), after every 10 samples (CCB), and end of run (CCB) | <= CRQL | Immed. after calib.(ICB), after every 10 samples (CCB), and end of run (CCB) | <1/2 RL | Immed. after calib., after every 10 samples, and end of run (ICB/CCB) |
| Low Level Check Std. (CRI/CRDL/CRQL) | --- | --- | +/- 20% | Daily after init. calib. For single pt. curve analysis | +/- 20% | Prior to analysis of samples unless multipoint (3+) curve w/ low std <=RL | --- | Every 20 samples. "CRI" at 2x the CRDL, not req. for Al, Ba, Ca, Fe, Mg, K, Na immediately followed by ICSA/ICSAB | +/- 30%. (+/- 50% for Sb, Pb, Tl) | Every 20 samples. "CRI" at the CRQL, not req. for Al, Ba, Ca, Fe, Mg, K, Na immediately followed by ICSA/ICSAB | None | --- |
| ICSA | --- | At beginning of analytical run | Absolute value of <2x MDL for non-spiked analytes | At beginning of analytical run | Absolute value of <2x MDL for non-spiked analytes | At beginning of analytical run | +/- 2x CRDL | At beginning and end of analytical run. Not greater than 20 samples per analysis run. | +/- 2x CRQL or +/-20% of true value | At beginning and end of analytical run. Not greater than 20 samples per analysis run. | --- | At beginning of analytical run |
| ICSAB | +/- 20% | At beginning of analytical run | +/- 20% | At beginning of analytical run | +/- 20% | At beginning of analytical run | +/- 20% | At beginning and end of analytical run. Not greater than 20 samples per analysis run. | +/- 2x CRQL or +/-20% of true value | At beginning and end of analytical run. Not greater than 20 samples per analysis run. | --- | At beginning of analytical run |

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| Study/QC | 6010B | | DOD-QSM (version 3, 2006) | | AFCEE (4.0.02) | | ILM 4.1 | | ILM 5.3 | | 200.7 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency | Criteria | Frequency |
| LCS (BS) | --- | --- | 80-120% | One per prep batch | 80-120% | One per prep batch | $H_2O$: 80-120% (except Ag and Sb). Soil: EPA Control limits | One per prep batch per matrix | $H_2O$: 80-120% (except Ag and Sb). Soil: EPA Control limits | One per prep batch per matrix | 85-115% | One per prep batch per matrix |
| Matrix Spike (M | 75-125% | One per prep batch per matrix | 80-120% | One per prep batch per matrix | $H_2O$: 80-120% (exc. Mo which is 79-120%. Soil: 80-120% (exc. Al which is 79-120% & Ag which is 75-120% | One MS/MSD per 20 samples | 75-125%, unless sample level >4x spike level | One per prep batch per matrix | 75-125%, unless sample level >4x spike level | One per prep batch per matrix | 70-130% | 10% of samples (MS/MSD per 20 samples) |
| Duplicate (D) | RPD <= 20% | One per prep batch per matrix | RPD <= 20% | One per prep batch per matrix | Matrix Spike:: $H_2O$: RPD <= 20%, Soil: <=30% | One MS/MSD per 20 samples | RPD <= 20% if analyte level >=5x CRDL. +/- CRDL, otherwise. | One per prep batch per matrix | RPD <= 20% if analyte level >=5x CRQL. +/-CRQL, otherwise. | One per prep batch per matrix | --- | --- |
| Serial Dilution | % Diff <= 10% | One per prep batch per matrix for a new or unusual matrix | % Diff <= 10%, if analyte conc. >50x MDL | One per prep batch per matrix | % Diff <= 10% | One per prep batch per matrix | % Diff <= 10%, if analyte conc. >50x IDL | One per prep batch per matrix | % Diff <= 10%, if analyte conc. >50x MDL | One per prep batch per matrix | % Diff <= 10%, if analyte conc. >50x MDL | --- |
| Post Digestion | 75-125% | One per prep batch per matrix for a new or unusual matrix | 80-120% | When dilution test fails or if an analyte's conc. for all samples in a batch is <50x MDL | 75-125% | When dilution test fails or if an analyte's conc. for all samples in a batch is <50x MDL | 75-125%, unless sample level >4x spike level | Only if MS fails (except Ag) | 75-125%, unless sample level >4x spike level | When the MS fails | 70-130% | --- |

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**Appendix III**

**Flow Chart**

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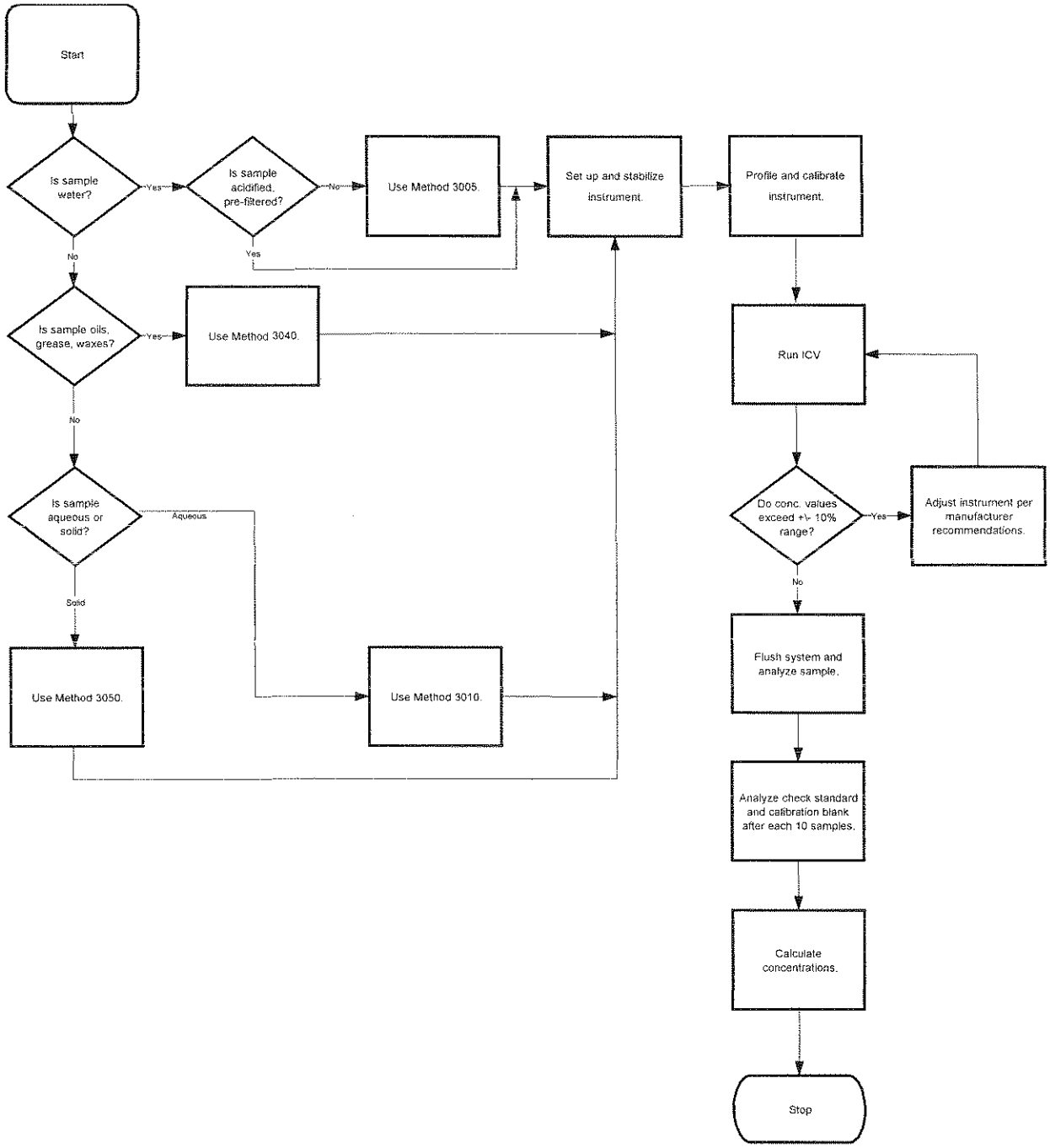

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## Appendix IV

### Sample Handling and Preservation

**Metals** (except hexavalent chromium and mercury):

| Measurement | Digestion Volume Required. | Collection Volume | Preservation/Holding Time |
|---|---|---|---|
| Total recoverable | 100 mL | 600 mL | $HNO_3$ to pH <2<br>6 months |
| Dissolved | 100 mL | 600 mL | Filter on site; $HNO_3$ to pH <2<br>6 months |
| Suspended | 100 mL | 600 mL | Filter on site<br>6 months |
| Total | 100 mL | 600 mL | $HNO_3$ to pH <2<br>6 months |

Solid samples should be at least 200 g and usually require no preservation other than storing at 4°C until analyzed. Either plastic or glass containers may be used for sample collection.

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## Appendix V

## Analytical Sequence

Standard 0
Standard 1
Standard 2
Standard 3
Standard 4
Standard 5
ICV
ICB
CRDL
ICSAI
ICSABI
CCV
CCB
Sample #1
Sample #1D
Sample #1S
Sample #1L
Sample #1P
Sample #2
Sample #3
Sample #4
Sample #5
Sample #6
CCV
CCB

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## Appendix VI

### Routine Reporting Limits

| Element | Water RL (µg/L) | Soil RL (mg/Kg) |
|---|---|---|
| Ag | 10 | 1.0 |
| Al | 500 | 50 |
| As | 20 | 2.0 |
| B | 1000 | 100 |
| Ba | 200 | 20 |
| Be | 5 | 0.5 |
| Ca | 5000 | 500 |
| Cd | 5 | 1.0 |
| Co | 50 | 5.0 |
| Cr | 10 | 1.0 |
| Cu | 25 | 2.5 |
| Fe | 100 | 10 |
| K | 5000 | 500 |
| Mg | 5000 | 500 |
| Mn | 15 | 1.5 |
| Mo | 20 | 2.0 |
| Na | 5000 | 50 |
| Ni | 40 | 4.0 |
| Sb | 60 | 6.0 |
| Pb | 10 | 1.0 |
| Se | 10 | 1.0 |
| Sn | 100 | 10 |
| Sr | 5 | 0.5 |
| Tl | 20 | 2.0 |
| V | 50 | 5.0 |
| Zn | 40 | 4.0 |

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## Appendix VII

### Estimated MDLs

| Element | Water MDL (µg/L) | Soil MDL (mg/Kg) |
|---|---|---|
| Ag | 0.4 | 0.04 |
| Al | 50 | 6 |
| As | 4 | 0.3 |
| B | 1.5 | 0.08 |
| Ba | 3 | 0.05 |
| Be | 0.4 | 0.03 |
| Ca | 100 | 3.2 |
| Cd | 0.4 | 0.2 |
| Co | 0.6 | 0.07 |
| Cr | 1 | 0.04 |
| Cu | 3.5 | 0.05 |
| Fe | 20 | 1.5 |
| K | 85 | 2 |
| Mg | 16 | 2.5 |
| Mn | 1 | 0.01 |
| Mo | 2 | 0.1 |
| Na | 175 | 15 |
| Ni | 1 | 0.1 |
| Sb | 1.5 | 0.1 |
| Pb | 2 | 0.2 |
| Se | 2.5 | 0.4 |
| Sn | 3 | 0.4 |
| Sr | 1 | 0.01 |
| Tl | 5 | 0.4 |
| V | 0.8 | 0.5 |
| Zn | 6.5 | 0.2 |

*Note: MDLs may vary slightly between digestion methods. The MDLs listed are estimated.*

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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8000

Title: **Determination of Retention Time Windows**

Revision history:

| Number | Date |
|---|---|
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| 2 | 06/22/00 |
| 3 | 06/20/02 |
| 4 | 07/28/04 |
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Revised by: Gail Rossidivito Date: 5/7/07
Gail Rossidivito, Senior Chemist

Approved by: Hugh Prentice Date: 5/7/07
Hugh Prentice, Laboratory Director

Approved by: Harry Romberg Date: 5/4/07
Harry Romberg, QA Officer

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## 1. Introduction and Scope

1.1 Method Description

1.1.1 Retention time windows are crucial to the identification of target compounds and surrogates. Absolute retention times are used for compound identification in all GC, HPLC, and ion chromatographic determinations. Retention time windows are established to compensate for minor shifts in absolute retention times as a result of normal chromatographic variability.

1.1.2 The width of the retention time windows should be such that the occurrence of both false positives and false negatives is minimized. Retention time windows that are too narrow can result in false negatives or may cause unnecessary reanalysis of sample extracts when surrogate or matrix spike compounds cannot be correctly identified. Conversely, retention time windows that are too wide may result in false positive results that cannot be confirmed by secondary column analysis or other methods.

1.1.3 This procedure describes the methodology used to establish retention time windows for chromatographic methods. It is based on the practice outlined in SW846, Method 8000B. Standards are analyzed over a time period of no less than 72 hours. Injections made over a period of less than 72 hours may result in retention time windows that are unrealistically small. The measured retention times of the standards are tabulated and a statistical measure of retention time stability is computed. This measure is then used to set the retention time window half-width used for analyte identification.

1.1.4 In general, a retention time window study is performed during method validation. The retention time windows thus determined are subsequently used for analyte identification during sample analysis. This method is intended to apply to all chromatographic methods performed at Laucks that do not employ a mass spectrometer as the detector (gas chromatography, HPLC, and ion chromatography).

## 2. Definition of Terms and Acronyms

2.1.1 **Retention Time Window (RTW)** –. The +/- value, which is applied to the ICV to establish the time range used to make tentative compound identifications. The retention time window half-width is set at 3 times the calculated standard deviation. The width is displayed in minutes or fractions of minutes.

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## 3. Equipment list and standards

3.1 Equipment

3.1.1 Chromatographic system

3.1.1.1 The system that will be used for the analysis of samples or sample extracts must be used in the determination of retention time windows. Ensure that the chromatographic system is operating reliably and that conditions have been optimized for the target analytes and surrogate compounds to be determined in the method.

3.2 Standards

3.2.1 Calibration standards required by the analytical method.

## 4. Safety precautions and waste disposal

4.1 Safety precautions

4.1.1 Standards, samples, and sample solutions

4.1.1.1 Handle all standards, samples, and solutions as if they are hazardous substances.

4.1.2 Instrument operation

4.1.2.1 Refer to the instrument manufacturer's manual for routine instrument precautions. Routine precautions include an awareness of the moving parts on the instrument being used. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

4.1.3 Electrical shock

4.1.3.1 Most analytical instruments present the possibility of electrical shock. All precautions should be taken to prevent electrical shock. This includes ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

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4.2 Waste disposal

4.2.1 All waste disposal precautions and procedures are detailed in the appropriate analysis SOP.

4.2.2 Waste segregation and disposal from the point of collection is further covered in the appropriate Laucks SOP.

## 5. Calibration and quality control

5.1 Calibration

5.1.1 Calibrate the instrument as described in the appropriate analytical method SOP. Before establishing retention time windows make sure that the chromatographic system is operating under optimal conditions.

## 6. Operation procedures

6.1 Data collection

6.1.1 For each standard analyzed over the course of at least one analytical sequence (no less than 72 hours long) tabulate the retention times for all target analytes and surrogates.

**Note**
A single analytical sequence is the minimum requirement. In most cases it is advisable to collect data over additional sequences in order to capture a data set of retention times that more closely models real world operating conditions.

6.1.2 The standards must bracket samples (or sample extracts) just as in a normal sample analysis sequence. Collect data for a minimum of 3 standards in each sequence. For multi-peak analytes such as Aroclors, tabulate the same peaks used for routine compound identification.

6.1.3 Record the retention times to 3 decimal places.

6.2 Calculations

6.2.1 Compute the mean and standard deviation of the measured retention times for each compound using the following equations.

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6.2.2 Mean

$$\bar{t} = \frac{\sum_{1}^{n} t_i}{n}$$

6.2.3 Standard deviation

$$SD = \sqrt{\frac{\sum_{i=1}^{n} \left(t_i - \bar{t}\right)^2}{n - 1}}$$

Where:
$t_i$=measured retention time
$\bar{t}$=average retention time
$n$=number of measurements

6.2.4 Retention time window (RTW)

$$\text{RTW} = \bar{t} \pm 3 * SD$$

6.2.5 Frequency of RTW determination

6.2.5.1 This study must be repeated whenever there is a major change to the method such as a new column, a new instrument temperature program, a new gradient program, major instrument overhaul, etc. It is desirable that multiple instruments running the same method have identical RTWs. However, this must be verified experimentally.

6.2.6 Constraints on the experimental determination of RTWs

6.2.6.1 RTW too small

6.2.6.1.1 In spite of the effort to mimic real world operating conditions by performing this study using a real analytical sequence, the RTWs may be unrealistically small, even 0.000 minutes. In this case use one of the following methodologies to administratively set the RTWs. When applying one of the administrative methodologies, the analyst's judgment weighs heavily. The desired result is that the RTWs be set such that the window half-width is sufficient to ensure that the chance for both false negatives and false positives is minimized.

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6.2.6.1.2 Method 1

Collect additional data and re-compute the RTWs.

**Note:**
Collecting data over more than one 72-hour analytical sequence should preclude a necessity to use Method 1.

6.2.6.1.3 Method 2

Set the RTW using the following guidelines.

| Run Type | RTW half-width, minutes |
|---|---|
| Narrow bore, megabore capillary GC | 0.03 |
| HPLC | 0.15 for analytes with RT to 15 min, 0.20 thereafter (see following notes) |
| Ion chromatography | 0.15 for analytes with RT to 15 min, 0.20 thereafter (see following notes) |

**Notes:**
In the determination of pesticides/PCBs using the EPA CLP Statement of Work, the RTWs are fixed. This methodology has been adopted by the laboratory for the determination of pesticides/PCBs by SW-846 Methods.

The default retention time window half-width (0.03 minutes) that is called for in SW-846 may be unrealistically small for HPLC determinations. The numbers listed in the table above are based on Laucks experience with these methods. In most cases, there will be sufficient retention time variation for HPLC analytes that this option will not be necessary.

This method is also implemented for ion chromatography. Ion chromatography is not addressed in SW-846, method 8000B.

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6.2.6.2 RTW too large

6.2.6.2.1 If, in the analyst's judgment, the compound RTW is too large then compute a pooled standard deviation using the following equation. Using this pooled estimate, re-compute the RTW as ±3 times the pooled estimate.

$$S_{pooled} = \sqrt{\frac{\sum_{1}^{k} s_k^2 (n_k - 1)}{\sum_{1}^{k} (n_k - 1)}}$$

k=k*th* set

$s_k^2$ =variance of k*th* set

$n_k$ =replicates in k*th set*

## 7. Application to routine analytical sequences

7.1 Setting the RTW for each analytical sequence

7.1.1 The analytical method for each SOP covers this in more detail, but the general methodology is as follows. After the analysis of the initial calibration verification standard, the RTWs for analyte identification in that analytical sequence are reset using the retention times of the mid-point calibration standard as the center of the window and the RTWs calculated above.

7.2 Analyst discretion in analyte identification

7.2.1 In addition to the RTW established for analyte identification, the judgment of the analyst also weighs heavily in the interpretation of chromatograms. Sample-specific effects can alter the observed retention times of target analytes in samples and sample extracts. In such cases, it is acceptable for the analyst to determine that the target analytes are outside the established RTWs.

7.2.2 Some techniques that are used for such identifications are retention time ratios of target analytes to surrogate compounds, observation of retention times of target analytes in MS/MSD samples, or re-analysis of sample extracts after spiking the extract with target analytes and observing an increase in peak response. Whatever methodology is used it must be completely documented in the narrative comments for the sample set.

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7.3 Corrective actions

7.3.1 The surrogate compounds added to each sample, blank, QC sample, and calibration standard are used to monitor retention time shifts. If a surrogate compound's retention time falls outside the expected RTW then the analyst must determine the cause and correct the problem before proceeding with further determinations.

**Note:**
In some cases sample-specific matrix effects may result in uncorrectable retention time shifts of surrogate compounds. These effects and corrective action taken must be documented in the narrative.

7.3.2 The retention time for all target analytes in continuing calibration standards must fall within the established windows. If retention times drift outside the established windows then perform instrument maintenance, analyze a new initial calibration verification standard, and reset the RTW centers.

## 8. Reports

8.1 Original data

8.1.1 The appropriate analytical department will retain the original files of all RTW studies.

8.2 Working copies

8.2.1 Summarized, tabulated retention time window results will be maintained in the appropriate chromatography laboratory, on an instrument specific basis. On a project specific basis and by request only, these summarized, tabulated RTW results will be provided in the case narrative report.

## 9. References:

- John Keenan Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, 1989
- USEPA, *Test Methods for Evaluating Solid Waste*, SW-846, 8000B, various analytical methods.
- USEPA *Statement of Work for Organics Analysis*, OLM04.2

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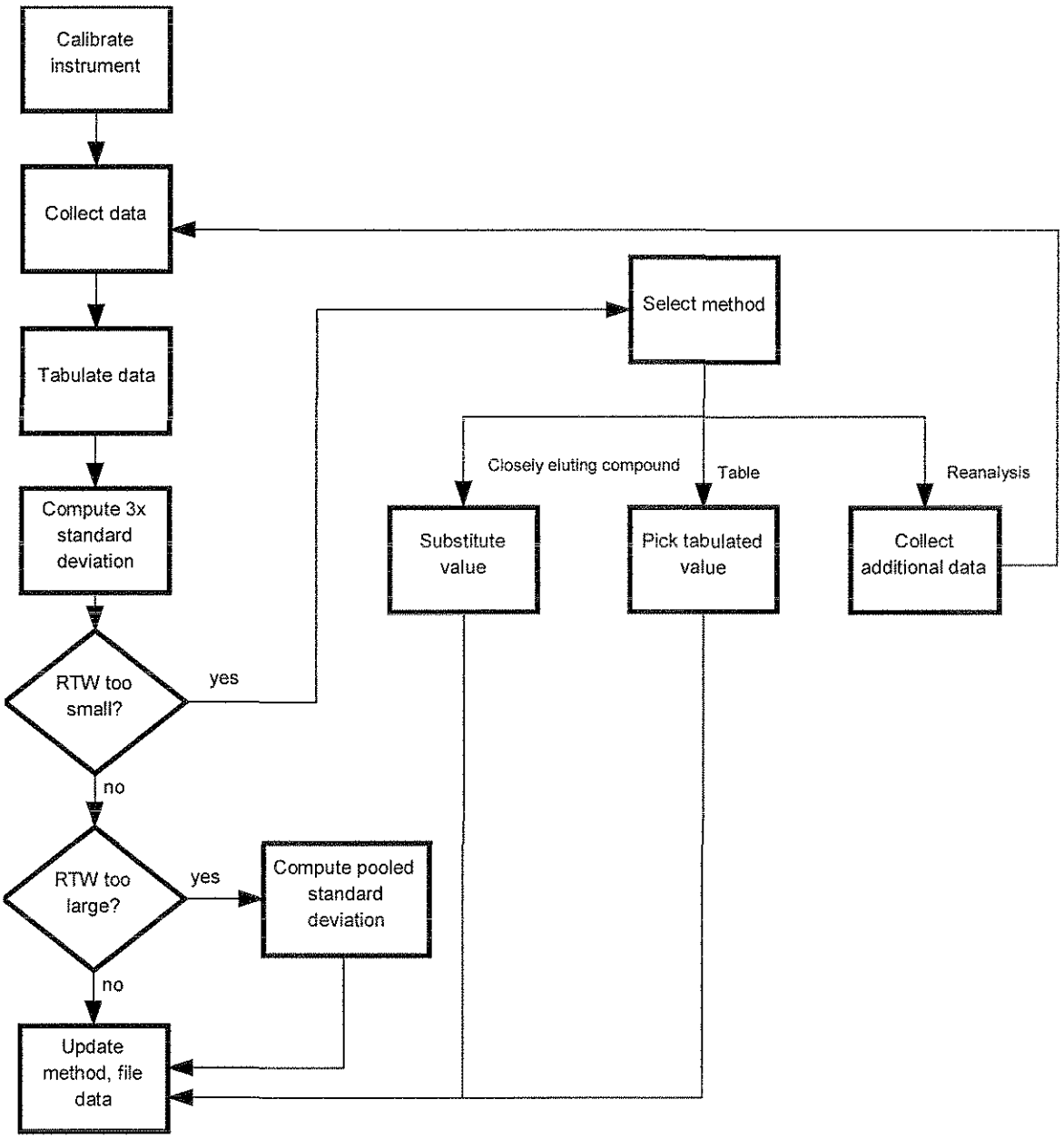
# Appendix 1

## LTL-8000 Flow Chart



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LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8084

Title: **Analysis of Organochlorine Pesticides and PCBs by SW846 Methods 8081B and 8082A**

Revision history:

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|---|---|
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| 2 | 4/29/99 |
| 3 | 4/27/00 |
| 4 | 9/11/01 |
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| 6 | 05/30/02 |
| 7 | 09/25/02 |
| 8 | 04/06/05 |
| 9 | 05/30/07 |

Revised by: Monica Carr, Technical Reviewer Date: 5/31/07

Approved by: Hugh Prentice, Laboratory Director Date: 6/1/07

Approved by: Harry Romberg, QA Officer Date: 5/31/07

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# 1 Introduction and Scope

## 1.1 Method Description

1.1.1 This SOP describes the procedures and specifications for instrumental analysis of various organochlorine pesticides and polychlorinated biphenyls (PCBs) in water and soil following SW846 Methods 8081B and 8082A. Analysis is performed by gas chromatography using a single injection port and splitting into dual GC columns with electron-capture detectors. This system provides quantitation and confirmation of pesticides and Aroclors (PCBs) from a single injection. The following table lists the compounds that may be determined by these methods. Additional compounds, listed in Method 8081B, may also be determined. Technical Chlordane will not be reported unless specified by the client.

| **SW8081B ROUTINE COMPOUNDS** | |
|---|---|
| alpha-BHC | beta-BHC |
| delta-BHC | gamma-BHC (Lindane) |
| Heptachlor | Aldrin |
| Heptachlor epoxide | Endosulfan I |
| Dieldrin | 4,4'-DDE |
| Endrin | Endosulfan II |
| 4,4'-DDD | Endosulfan sulfate |
| 4,4'-DDT | Methoxychlor |
| Endrin aldehyde | Endrin ketone |
| alpha-Chlordane | gamma-chlordane |
| Toxaphene | |
| **SW8081B OPTIONAL COMPOUNDS** | |
| Chlordane (not otherwise specified) | Isodrin |
| Simazine | Atrazine |

| **SW8082A COMPOUNDS** | |
|---|---|
| Aroclor-1016 | Aroclor-1221 |
| Aroclor-1232 | Aroclor-1242 |
| Aroclor-1248 | Aroclor-1254 |
| Aroclor-1260 | |
| SW8082A OPTIONAL COMPOUNDS (Required by DoD) | |
| Aroclor-1262 | Aroclor-1268 |

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the use of gas chromatography and in the interpretation of chromatograms. Each analyst performing this method must have demonstrated the ability to perform the described

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chromatographic analysis and/or data interpretation.

1.1.3 In some instances, samples being analyzed for PCBs will require sulfuric acid cleanup. In instances where samples are being analyzed for both pesticides and PCBs, an aliquot is separated prior to the sulfuric acid cleanup step, and is analyzed for pesticides only. The separation of aliquots prior to this cleanup will prevent the potential loss of target pesticide compounds.

1.2 Method Deviations

1.2.1 The analyte list is based on a combination of Method 8081B and 8082A analytes. Additional analytes are only added if requested by the client(s).

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Samples are normally collected in glass containers with Teflon-lined caps. All samples and sample extracts are stored at 4°C±2°C. Water samples must be extracted within 7 days of sample collection, soil samples within 14 days of sample collection. All extracts must be analyzed with 40 days of sample preparation.

1.4 Definition of Terms

1.4.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

| | |
|---|---|
| Batch Identifier | A number given to each preparation or analysis group which uniquely identifies that batch. This number is generally the blank ID for preparation batches and either a sequence number for organic analyses or an analysis number that is similar to the blank ID, only preceded by an "A" rather than a "B" for inorganic batches. The preparation batch IDs are discussed in other documentation. |
| Blank spike | A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts of target analytes and surrogates are added each time sample extracts are prepared. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard. |
| CCV | Continuing calibration verification. This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency during the analysis sequence to verify that the instrument has remained in calibration. |

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| | |
|---|---|
| CF | Calibration factor. The ratio of analyte instrument response to nanograms injected. This term is defined in the same way in both the CLP contract and SW 846. |
| CLP | Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition. |
| DCC | Daily calibration check. This is the first standard analyzed in the sequence. |
| DIW | Deionized water. Lab reagent water. Organic-free water. Since the systems used to provide DIW at Laucks all contain carbon-polishing filters, they are capable of providing organic-free water for use in method blanks and blank spikes. |
| IBLK | Instrument blank. This term is borrowed from CLP. Blank solvent containing the method surrogates is injected into the instrument to monitor for carry over between sample extract injections. |
| ICV | Initial calibration verification. It is a standard that is injected at the start of each QC period that is compared to the initial multi-point calibration to determine whether the instrument is still in calibration. |
| IDL | Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels. |
| MDL | Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks. |
| MDL standard | Method detection limit standard. A standard prepared so that the concentrations of the target analytes are in the range of 1x to 4x the empirically determined MDLs on an extract/digest basis. This standard is used to verify that the instrument is capable of detecting the target analytes on an ongoing basis. |

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| | |
|---|---|
| PQL or Reporting Limit | Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set. |
| QC check standard | Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike. |
| QC period | Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP. |
| Reporting Limit (RL) | The reporting limit is defined by the low level standard, also referred to as the practical quantitation limit (PQL). |
| RSD or %RSD | Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another. |
| RT, Retention time | The time (in minutes) at which a target analyte elutes from a chromatography column. |
| RT window | Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications. |
| Sequence | A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period. |
| SRM | Standard Reference Material - A material containing known quantities of target analytes in a homogeneous matrix which approximates the matrix of the samples being analyzed. It is used to establish that the analytical process is in control. |

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## 2 Equipment List, Maintenance, and Standards

2.1 Chromatographic and Data System

2.1.1 This analysis requires a gas chromatograph with a programmable oven, heated injection port, dual electron-capture detectors, autosampler and an electronic data-acquisition system.

2.1.2 Equipment list:

- Gas chromatograph (Hewlett-Packard 5890, 5890A, 6890 w/µECDs) or equivalent
- Autosampler (Hewlett-Packard 7673A, 7683) or equivalent
- Electron-capture detectors (Hewlett-Packard) or equivalent
- Capillary columns (J&W DB5, DB608, 30m x 0.45mm, or equivalent)
- 0.45mm megabore, J&W DB-XLB, DB35ms 30m x 0.32mm, or equivalent)
- 5% methane/95% argon detector make-up gas,
- Helium carrier gas
- EZChrom Data Acquisition System version 6.6, Target Analytical Software for NT
- Operating System version 4.02, Pentium PCs, or equivalent.

2.1.3 Maintenance

2.1.1 In order to prevent/reduce endrin breakdown, the following steps are used:

1) use clean and silanized Agilent liners, (splitless, single-taper liners for the 6890)
2) use only gold seals
3) reduce the injection port temperature e.g., from 250°C to 205°C
4) clean inlet with hexane and acetone
5) prime inlet with really high concentration standard
6) make sure syringe does not touch glass wool.

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2.2 Standards

2.2.1 Commercially prepared, certified stock standards are used to prepare working solutions for all surrogates, calibration mixes, and spike mixes. The calibration mix (INDAB) contains all of the individual pesticide analytes. All individual pesticides are spiked. Aroclors 1016 and 1260 are spiked. Stock standards must be replaced by the manufacturers expiration date.

2.2.2 Calibration standards are prepared at different concentration levels by dilution of the stock standards with hexane (see table below). The standards must be replaced after 6 months or by the expiration date of the material they were made from, whichever is earlier.

2.2.3 Multicomponent analyte calibration standards, with the exception of Aroclors 1016 and 1260 (combined are defined as Aroclor 1660), are prepared at a concentration of 1.0 µg/ml, by dilution of commercially purchased stock solutions in hexane (see table below). Toxaphene is made up to a concentration of 0.5 µg/ml and chlordane is made up to a concentration of 0.1 µg/ml by dilution of commercially purchased stock solutions in hexane. Standards of the Aroclors 1016 and 1260 are combined in a mixture and prepared at five concentration levels: 0.5 µg/ml, 1.0 µg/ml, 0.25 µg/ml, 0.5 µg/ml, and 0.75 µg/ml. These must be replaced after 6 months or by the parent expiration date, whichever is earlier.

| **Standard level** | **INDAB Mix µg/mL** | **AR1660 µg/mL** | **All other Aroclors µg/mL** | **Toxaphene µg/mL** | **Chlordane µg/mL** |
|---|---|---|---|---|---|
| 1 | 0.002-0.004 | 0.05 | N/A | 0.005 | 0.025 |
| 2 | 0.0025-0.005 | 0.10 | N/A | N/A | N/A |
| 3 | 0.005-0.01 | 0.25 | 0.5 | N/A | N/A |
| 4 | 0.008-0.016 | 0.5 | N/A | N/A | N/A |
| 5 | 0.02-0.4 | 0.75 | N/A | N/A | N/A |
| 6 | 0.04-0.8 | N/A | N/A | N/A | N/A |
| 7 | 0.04-0.08 | | | | |

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2.3 Second Source Standards

2.3.1 The second source Aroclor standards are purchased from both Supleco and Ultra Scientific. (All Aroclors require second source standards.) Current Supelco catalog numbers are 9-0129R, 9-0123R The Ultra catalog number is 091405. The concentration of these standards are 1000 µg/mL. The intermediate solution is made by taking 10 µL into 10 mL hexane. The second source standard concentration is varied at the instrument. Refer to the table above to view the range of concentrations for Aroclors 1016 and 1260. These are the same concentration used for all Aroclors. The working solution is good for 6 months or the parent's expiration, whichever is sooner.

2.3.2 The second source pesticide standard is purchased from Ultra Scientific. The catalog number is PPM-808C. The standard concentration is 1000 µg/mL. This standard is made up like the CCV standard as explained above. Refer to section 1.1.1. The solutions are good for 6 months or until the parent's expiration date, whichever is sooner.

2.3.3 A performance evaluation mixture (PEM) is prepared from a purchased solution of 4,4'-DDT, and endrin in hexane.

2.3.4 A surrogate mix is prepared from separate solutions that contain 2,4,5,6-tetrachloro-meta-xylene (TCMX) and decachlorobiphenyl (DCB). These standards are made in hexane and added to all standard mixes (before dilution if applicable).

2.3.5 Refer to SOP LTL-1013 (Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials) for detailed instructions on standards preparation and storage.

2.3.6 Appendix I details the compounds and concentrations contained in all solutions.

## 3 Safety precautions and Waste Disposal

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they contain hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

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3.1.5 The electron-capture detectors used in this analysis contain a radioactive source and should not be opened or otherwise tampered with.

3.2 Waste disposal

3.2.1 Solvent wastes generated in using this procedure (expired standards, old sample extracts, rinsates, etc.) should be emptied into the solvent waste container in the fume hood.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

## 4 Calibration and Quality Control

4.1 QA Requirements and Corrective Action

4.1.1 Detailed in the following sections are applicable QA requirements and subsequent corrective actions to be applied to this analysis. A summary of these requirements can be found in Appendix IV.

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on MDLs, located in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies are performed on every Aroclor. Aroclors 1016 and 1260 can be combined (they are referred to as 1660.) In addition, an Aroclor MDL study is performed on every instrument for each matrix or separate prep.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.3.2 The precision of spike recoveries must meet or exceed the criteria tabulated in SW 846.

4.4 Retention Time Windows

4.4.1 Retention time window studies are conducted following the Laucks SOP on Establishing RTWs and the procedure detailed in SW 846, Method 8000B, which defines a window for each single and multi- component analyte based on the retention times taken from three standard injections over a 72 hour period.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are

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made, whenever the chromatographic method is significantly modified, or whenever a column is replaced.

4.4.3 The calculated retention time window half widths are typically unrealistic values because the standard deviation is routinely zero. Therefore RT windows have been administratively set at the values listed in OLM04.2 for megabore analyses (see table below) and at ±0.03 for capillary analyses. These values are typically wider than 3 times the standard deviation as determined above, but are more realistic. The adminsitratively set widths may not be narrower than the statistically derived retention time half widths.

| Analyte | Megabore | Capillary |
|---|---|---|
| alpha-BHC | ±0.05 | ±0.03 |
| beta-BHC | ±0.05 | ±0.03 |
| gamma-BHC (Lindane) | ±0.05 | ±0.03 |
| delta-BHC | ±0.05 | ±0.03 |
| Heptachlor | ±0.05 | ±0.03 |
| Aldrin | ±0.05 | ±0.03 |
| alpha-Chlordane | ±0.07 | ±0.03 |
| gamma-Chlordane | ±0.07 | ±0.03 |
| Heptachlor epoxide | ±0.07 | ±0.03 |
| Dieldrin | ±0.07 | ±0.03 |
| Endrin | ±0.07 | ±0.03 |
| Endrin aldehyde | ±0.07 | ±0.03 |
| Endrin ketone | ±0.07 | ±0.03 |
| 4,4'-DDD | ±0.07 | ±0.03 |
| 4,4'-DDE | ±0.07 | ±0.03 |
| 4,4'-DDT | ±0.07 | ±0.03 |
| Endosulfan I | ±0.07 | ±0.03 |
| Endosulfan II | ±0.07 | ±0.03 |
| Endosulfan sulfate | ±0.07 | ±0.03 |
| Methoxychlor | ±0.07 | ±0.03 |
| Tetrachloro-m-xylene | ±0.07 | ±0.03 |
| Decachlorobiphenyl | ±0.10 | ±0.03 |
| Aroclors | ±0.07 | ±0.03 |
| Toxaphene | ±0.07 | ±0.03 |

4.5 Breakdown Evaluation (only required when analyzing for pesticides)

4.5.1 At the beginning of each analysis sequence a performance evaluation mixture (PEM) must also be analyzed. This is a separate solution containing 4,4'-DDT and endrin, and is examined for the breakdown products of these analytes (4,4'-DDD, 4,4'-DDE, endrin aldehyde, endrin ketone) which indicate the need for GC system maintenance. The

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percent breakdown must be less than 15% for both Endrin and 4,4'-DDT.

4.6 Initial Multi-Point Calibration for Pesticides and PCBs

4.6.1 SW 846 allows the use of both linear and non-linear models for the calibration data. The option for non-linear calibration may be necessary to achieve low detection limits or to address specific instrumental techniques. Non-linear calibrations are not allowed for the Aroclors.

4.6.2 Analyze single component pesticide standards using at least 5 different concentration levels for linear calibrations. The lowest concentration should be near, but above, the method reporting limit or PQL. The highest concentration should define the upper usable working range of the detector.

4.6.3 Analyze multi-component pesticides at one concentration level. Initial calibration of Aroclor 1016/1260 is performed using a minimum of five different concentration levels. The more commonly found Aroclors (Aroclors 1248 and 1254) are calibrated using three different calibration levels. The detection of any Aroclor in a sample requires a minimum of three calibration points, followed by re-analysis of the sample extract. Extracts containing toxaphene and chlordane are extremely rare so an initial single point calibration is employed. Single standards of Aroclors, toxaphene, and chlordane are required to aid the analyst in pattern recognition and are also used to determine the initial calibration factor for each analyte. If the sample extract contains toxaphene or chlordane above the reporting limit, a three-point calibration will be analyzed for the target analyte followed by re-analysis of the sample extract. The three point calibration points will be diluted from the stock standard and must be at concentrations that will bracket the concentration of the sample. This three-point calibration method will be used to determine the final calibration factor for the target analyte. The CF is calculated by using a minimum of three to five peaks for the multi-component pesticide and PCB standards.

4.6.4 The choice of a specific calibration model should be made in one of two ways. The first is to begin with the simplest approach, the linear model through the origin, and progressing through the other options until the calibration acceptance criteria are met, or until the analyte is best represented by a calibration model. The second approach is to use *a priori* knowledge of the detector response to choose the calibration model. Such knowledge may come from previous experience, knowledge of physics of the detector, or specific manufacturer's recommendations.

4.7 Linear Calibration

4.7.1 External standard initial calibration data is evaluated by determining the %RSD of the calibration factors.

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4.7.2 CFs are calculated using the equation:

$$CF = \frac{\text{response}}{\text{ng injected}}$$

4.7.3 The calculated CFs are tabulated and the %RSD calculated. There are no compound-specific criteria. All %RSDs must be within 15% for each analyte plus recovery of all analytes must be within 80-120%.

4.7.4 Corrective action

4.7.4.1 If the criteria are not met, the instrument must be re-calibrated or if appropriate, an alternative calibration model employed. The appropriate calibration model can be selected through the use of the GC initial calibration exceptions report. This is available as a template in Microsoft Excel.

4.8 Non-linear Calibration

4.8.1 In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model may be employed for pesticides. (Non-linear calibrations are not allowed for the Aroclors.

4.8.2 When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen should have no more than four parameters, i.e., if the model is polynomial, it may be no more than third order, as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

4.8.3 The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches. A quadratic (second order) requires a minimum of six standards, and a third order polynomial requires seven standards. Do not force the line through the origin, i.e., do not set the intercept as 0, and do not include the origin (0,0) as a calibration point. The $r^2$ value must be $\geq 0.990$.

4.8.4 Whichever option is employed, a surrogate concentration must fall within the calibration range.

4.8.4.1 A set of three to five major peaks is selected for each multicomponent analyte. These should be characteristic of the multicomponent analyte in question. Retention time windows and calibration factors are generated for each of the peaks chosen.

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4.8.5 Corrective action

4.8.5.1 If the criteria are not met, the instrument must be re-calibrated.

4.9 Second Source Standards (Initial Calibration Verification-ICV)

4.9.1 A second source standard containing pesticides and all Aroclors must be analyzed after each initial calibration. The standard should be prepped at a concentration at varied concentration levels. The %D for each second source standard must be within 20%.

4.9.2 Corrective action

4.9.2.1 If the criteria are not met, the second source standard is reanalyzed. Alternatively, a standard from an additional source may be required if there continues to be differences > 15%.

4.10 Daily Calibration Check

4.10.1 Concentration and/or CF Criteria

4.10.1.1 At the beginning of an analysis sequence analyze a mid-range calibration standard. The computed calibration factor (CF) or concentration measurement must meet the criteria detailed below.

4.10.1.2 Using the appropriate calculation technique (average CF ) compute either CFs or concentration values. For linear calibrations the DCC standard can be verified by calculating either the percent difference or the percent drift.

4.10.1.2.1 The percent difference calculation compares the DCC CFs to the mean CFs from the initial multi-point calibration. The percent difference is calculated as follows:

$$\%D = \frac{Cv - \overline{CF}}{\overline{CF}} \times 100$$

where:

$C_V$ = Calibration Factor

$\overline{CF}$ = Mean Calibration Factor

4.10.1.2.2 The percent drift calculation compares the DCC calculated concentrations to the theoretical (or actual) concentration of the DCC standard. The percent drift is calculated as follows:

$$\%D = \frac{C_c - C_T}{C_T} \times 100$$

where:

$C_C$= Calculated Concentration
$C_T$ = Theoretical Concentration

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4.10.1.3 There are no compound-specific criteria. The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.10.2 Corrective action

4.10.2.1 If the DCC criteria are not met, no sample extracts can be analyzed. Perform system maintenance and re-check the DCC. If the criteria still cannot be met, the system must be recalibrated.

4.11 Updating Retention Time Windows

4.11.1 The retention time windows for compound identification are updated using the retention times for each target analyte in the mid-point calibration standard of the initial calibration as the center of the window and the previously determined retention time window half-width to establish the retention time range to be used for compound identification.

4.12 Instrument Blank

4.12.1 Criteria

4.12.1.1 Any sample that is suspected of containing high concentrations of target analytes should be followed by an IBLK or solvent rinse. This IBLK analysis is used only to make a judgment as to the possibility of carry-over into the sample extract immediately following the IBLK. Evaluation criteria are detailed below.

4.12.2 Corrective action

4.12.2.1 IBLKs used to monitor for possible carryover in high concentration extracts (those IBLKs optionally placed into the sequence following suspected high concentration extracts) are used to flag the possibility of analyte carryover into the following sample extract. The extract immediately following the out of control IBLK may need to be re-analyzed if there is a detectable amount at ½ the reporting limit of the analyte found in the IBLK.

4.13 Continuing Calibration Verification

4.13.1 The continuing calibration is verified each 12 hours by injection of a calibration standard. SW 846 requires that the calibration standard be injected at a frequency of not less than once every 20 samples (the frequency of every 10 samples is recommended to minimize the number of reinjection due to QC failure). Laucks analyzes the continuing calibration standards every 10 samples or every 12 hours, whichever is deemed appropriate by the analyst. In addition, this standard must be the last injection made in the analysis sequence.

4.13.2 Criteria

4.13.3 After 12 hours, a CCV standard is analyzed. The CF or concentration for each analyte is

calculated and the percent difference or percent drift is calculated as shown above.

4.13.4 The %D results should be within ±15% of the average CF or expected concentration from the initial calibration for each analyte, or averaged across all analytes in the continuing calibration standard.

4.13.5 The retention times for all target analytes must fall within the RT windows established by the mid-point of the initial calibration.

4.13.6 Corrective Action

4.13.7 Check calculations or perform instrument maintenance. To validate the quantitation of target analytes in analytical samples, the samples must be bracketed by in-control CCVs. However, CCV CFs can be outside the control limits as long as this was due to an increase in response and the corresponding samples contain no detectable levels of the target analyte for which the CF is out of control.

4.14 Method Blanks

4.14.1 Criteria

4.14.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples extracted at the same time or one blank every 20 samples which ever is more frequent. Any analyte response above ½ the reporting limit is considered out of control.

4.14.2 Corrective action

4.14.2.1 Corrective action may necessitate re-extraction of the sample set. For example, if an analyte were found in the blank but not in any of the associated samples then the sample group may not require re-extraction. In any event it is the laboratory's responsibility to ensure that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware leading to discrete artifacts and/or elevated baselines in the chromatograms be minimized. In the extreme case of chronic contamination, blanks may have to be analyzed from each stage of the sample processing to determine the contamination source so it can be eliminated. In all cases where blank contamination exceeds the control limit a narrative comment must be made which documents the corrective actions taken.

4.15 Blank Spikes (LCS)

4.15.1 Criteria

4.15.1.1 A blank spike follows the same protocol as with the matrix spike analysis except that the spiking solution is added to a method blank solution instead of an actual sample. A method blank with added analytes is a blank spike. A blank spike is the same as a QC check standard and LCS (Laboratory Control Sample). The single component pesticides and Aroclors 1016/1260 are spiked into the LCS. A subset of the single-

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component target analytes can be substituted for the full list if specified in project-specific contracts or workplans. The LCS and matrix spike should consist of the same components at the same concentration levels in order to accurately evaluate matrix interference in the MS/MSD samples.

4.15.1.2 Blank spike recoveries must meet the criteria specified in the LIMS.

4.15.2 Corrective action

4.15.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. Sample re- extraction and re-analysis would be triggered by an out of control blank spike only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

4.16 Matrix Spike

4.16.1 Criteria

4.16.1.1 A sample is chosen at random from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to extraction. It is required that a matrix spike analysis be performed with each extraction batch. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\%\text{Recovery} = \frac{MS - SA}{S} x100$$

where:
MS = concentration in spiked sample
SA = native concentration in unspiked sample
S = spike amount

4.16.1.2 The recovery criteria are detailed in the LIMS. In the instance that the native target analyte concentration is greater than 5x the spike concentration, the MS recovery control limits do not apply. In this case, treat the MS/MSD pair as duplicates and report them as such in the LIMS.

4.16.2 Corrective action

4.16.2.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action may involve recalculation, re-extraction, and/or reanalysis. This process should also look at the recovery of surrogate compounds in the MS sample and at the recovery of matrix spiking compounds from the extraction batch blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

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4.17 Matrix Spike Duplicate

4.17.1 Criteria

4.17.1.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$\%RPD = \frac{|\chi - \delta|}{(\chi + \delta)/2} x100$$

where:

$\chi$ = measured concentration for MS sample
$\delta$ = measured concentration for MSD sample

4.17.1.2 RPD control limits are detailed in the LIMS.

4.17.2 Corrective action

4.17.2.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

4.18 Surrogate Recovery

4.18.1 Criteria

4.18.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor for potential sample processing errors and matrix effects. Surrogate compound recoveries are calculated as follows:

$$\%\ \text{recovery} = \frac{S_m \times 100}{S_a}$$

where:

$S_m$ = concentration of surrogate measured in extract
$S_a$ = concentration of surrogate added

4.18.1.2 Detailed surrogate recovery control limits are tabulated in the LIMS.

4.18.2 Corrective Action

4.18.2.1 Check calculations for possible error. Low surrogate recoveries are greater potential

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indicators of poor method performance than high surrogate recovery since non-GC/MS methods cannot separate co-eluting interferences. Hence corrective action is not required for high surrogate recoveries. If TCMX and DCB surrogates are used in the PCB analysis when only DCB is required, both surrogates must be evaluated and will require corrective action for out of control recoveries.

4.18.2.2 Low surrogate recoveries in the method blank may require that all the samples in the associated batch be re-extracted and re-analyzed. In any case, it is imperative to identify the problem associated with low recovery so that it can be corrected. It is a requirement that all out of control surrogate recoveries and the corrective action taken be discussed in the narrative.

## 5 Operation procedures

### 5.1 Chromatographic Conditions

5.1.1 The following general operating parameters are used on gas chromatographs to perform this method utilizing megabore (0.45 mm) columns:

| | |
|---|---|
| Carrier gas: | helium |
| Column flow: | 8 mL/min |
| Make-up gas: | argon/methane (95%/5% High Purity grade) |
| Make-up flow: | 70 mL/min |
| Injector: | Grob-type, splitless |
| Injector temperature: | 205°C |
| Injection: | splitless |
| Injection volume: | 2µL (split - 1 µL per column) |
| Initial temperature: | 150°C |
| Initial hold time: | 0.5 min |
| Temperature ramp: | 4.5°C per min |
| Final temperature: | 275°C |
| Final hold time: | 10 min |
| EC detector temperature: | 350°C |

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5.1.2 The following general operating parameters are used on the 6890 gas chromatograph equipped with 0.32 mm columns for Pesticides:

| | |
|---|---|
| Carrier gas: | helium |
| Make-up gas: | argon/methane (95%/5% High Purity grade) |
| Column flow: | 3.6 mL/min |
| Make-up flow: | 30 mL/min |
| Pulsed pressure: | 55.0 psi |
| Pulsed time: | 0.60 min. |
| Purge flow: | 50.0 mL/min |
| Injector: | splitless, single-taper |
| Injector temperature: | 250°C |
| Injection: | splitless |
| Injection volume: | 1 µL (split – 0.5 µL per column) |
| Initial temperature: | 110°C |
| Initial hold time: | 0.5 min |
| Temperature ramp: | 15°C per min |
| Final temperature 1: | 320°C |
| Final time 1: | 2 min |

The following general operating parameters are used on the 6890 gas chromatograph equipped with 0.32 mm columns for PCBs:

| | |
|---|---|
| Carrier gas: | helium |
| Make-up gas: | argon/methane (95%/5% High Purity grade) |
| Column flow: | 2.6 mL/min |
| Make-up flow: | 35 mL/min |
| Pulsed pressure: | 55.0 psi |
| Pulsed time: | 0.60 min. |
| Purge flow: | 50.0 mL/min |
| Injector: | splitless, single-taper |
| Injector temperature: | 250°C |
| Injection: | splitless |
| Injection volume: | 1 µL (split – 0.5 µL per column) |
| Initial temperature: | 150°C |
| Initial hold time: | 0.5 min |
| Temperature ramp: | 25°C per min |
| Final temperature 1: | 250°C |
| Final time 1: | 1 min |
| Rate 2 | 15°C per min |
| Final temperature 2: | 340°C |
| Final time 2: | 2.00 min |
| EC detector temperature: | 350°C |

These GC conditions should be optimized for analyte separation and sensitivity with a

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particular pair of columns and detectors. Once optimized, the same conditions must be used for the analysis of all standards, samples, blanks and spikes.

5.2 Sample Analysis

5.2.1 Analysis sequence

5.2.1.1 See Appendix II for a detailed analysis injection sequence.

5.2.2 Compound Identification

5.2.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the column 1. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. (Second column confirmation is required for both pesticides and Aroclors.) Retention time windows are established as previously described and the absolute retention times are updated each QC period. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes.

5.2.2.3 Identification of multicomponent analytes occurs when the retention times and ratios of each of the multicomponent peaks present in the sample match the peaks chosen for the particular multicomponent analyte in the initial calibration. These same conditions must be met on both columns in order to confirm the presence of the multicomponent analyte. The experienced analyst's judgment weighs heavily in evaluating the patterns of multicomponent analytes with regards to weathering and matrix interferences.

5.2.3 Compound Quantitation

Target compound concentrations are calculated using the following equations:

5.2.3.1 Aqueous samples

5.2.3.1.1 The external standard equation, as expressed in SW 846 is:

$$\text{Concentration } (\mu g / L) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(V_s)}$$

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where:

$A_X$ = Response for the analyte in the extract, in area or height units.
$CF_m$ = Multi-point average CF
$V_i$ = Volume of extract injected, µL.
$D$ = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was diluted 100 µL to 1000 µL and subsequently diluted an additional 100 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
$V_t$ = Volume of total extract, µL.
$V_S$ = Initial sample size, mL.

The reported concentration for multicomponent analytes calculated is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.3.1.2 To report concentrations in alternate units, apply an appropriate factor:

$$\text{mg/L} = \mu\text{g/L} * 0.001$$

5.2.3.2 Non-aqueous samples

5.2.3.2.1 The results calculation for non-aqueous samples is very similar to that for aqueous samples. The only difference is the inclusion of a total solids term to calculate the dry weight equivalent of the initial sample size.

$$Conc.(\mu g / kg) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(W)(TS/100)}$$

where:
W = Weight of sample extracted or purged, grams.
TS = Total solids, percent.

The reported concentration for multicomponent analytes is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.4 Sample Dilution

5.2.4.1 If the responses in the sample chromatogram exceed the calibration range of the system, dilute the extract and reanalyze. The dilution should be made so that the concentration of the analyte requiring dilution is in the mid to upper calibration range.

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5.2.5 Sample Confirmation

5.2.5.1 When a dual column /dual detector GC technique is employed, a P flag indicates that calculated results from the two determinations differ by more than 40%. If the results from one column is significantly higher (40%) the chromatogram is checked for overlapping peaks, or irregular baseline integration. If no anomalies are discovered, the higher result is reported.

## 6 Reports

6.1 Data Packet Organization

6.1.1 See Appendix III for a checklist detailing data packet organization.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the quality control database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and an MS/MSD report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

| Code | Definition |
|---|---|
| U | The analyte of interest was not detected, to the reporting limit indicated. |
| B | The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The flag is applied without regard to the relative concentrations detected in the blank and sample. |
| J | The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate. |
| D | The value reported is derived from the analysis of a diluted sample or sample extract. |
| P | When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. If the results from one column is significantly higher (25%) the chromatogram is checked for overlapping peaks, or irregular baseline integration. If no anomalies are discovered, the higher result is reported in order to employ the conservative approach relative to protection of the environment. |
| E | The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate. |
| C | The target analyte's presence was confirmed by GC/MS. |

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6.4 Control Chart(s)

6.4.1.1 The recovery values for all individual pesticides, Aroclor 1016, Aroclor 1260, TCMX, and DCB in the Blank Spike are plotted on control charts.

## 7 References

1). U.S. EPA Contract Laboratory Program Contract, Exhibit D, Section III, March 1990

2). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method 8000B, Gas Chromatography, Revision 2, December 1996.

3). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Organochlorine Pesticides by Gas Chromatography, Method 8081B, Revision 2A, May 1999 .

4). U.S. EPA SW846 Test Methods for Evaluating Solid Waste, Method , Polychlorinated Biphenyls, (PCBs) by Gas Chromatography, Method 8082A, Revision 1A, May 1999 .

5). Department of Defense Quality Systems Manual for Environmental Laboratories, Department of Navy, Final Version 3, January 2006.

6). Technical Services Quality Assurance Project Plan (QAPP), HQ Air Force Center for Environmental Excellence, Final Version 4.0.01, May 2005.

7). National Environmental Laboratory Accreditation Program, Ninth NELAC Annual Meeting, June 5, 2003

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## APPENDIX I

**5890 Standard Solution Concentrations, µg/mL**

| Compound | STD1 | STD2 | STD3 | STD4 | STD5 |
|---|---|---|---|---|---|
| Aroclor-1016 | 0.1 | 0.25 | 0.5 | 0.75 | 1.0 |
| Aroclor-1221 | | | 0.5 | | |
| Aroclor-1232 | | | 0.5 | | |
| Aroclor-1242 | | | 0.5 | | |
| Aroclor-1248 | | | 0.5 | | |
| Aroclor-1254 | | | 0.5 | | |
| Aroclor-1260 | 0.1 | 0.25 | 0.5 | 0.75 | 1.0 |
| 2,4,5,6-tetrachloro-m-xylene | 0.01 | 0.025 | 0.05 | 0.075 | 0.1 |
| Decachlorobiphenyl | 0.01 | 0.025 | 0.05 | 0.075 | 0.1 |

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**6890 Standard Solution Concentrations, µg/L**

| Compound | STD1 | STD2 | STD3 | STD4 | STD5 |
|---|---|---|---|---|---|
| alpha-BHC | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| beta-BHC | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| delta-BHC | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| gamma-BHC (Lindane) | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| alpha-Chlordane | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| gamma-Chlordane | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| Heptachlor | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| Aldrin | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| Heptachlor epoxide | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| Endosulfan I | 4.0 | 8.0 | 20.0 | 40.0 | 80.0 |
| Dieldrin | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| 4,4'-DDE | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| Endrin | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| Endosulfan II | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| 4,4'-DDD | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| Endosulfan sulfate | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| 4,4'-DDT | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| Methoxychlor | 40.0 | 80.0 | 200 | 400 | 800 |
| Endrin aldehyde | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| Endrin ketone | 8.0 | 16.0 | 40.0 | 80.0 | 160.0 |
| Toxaphene | | | 200 | | |
| Aroclor-1016 | 50 | 100 | 250 | 500 | 750 |
| Aroclor-1221 | | | 250 | | |
| Aroclor-1232 | | | 250 | | |
| Aroclor-1242 | | | 250 | | |
| Aroclor-1248 | | | 250 | | |
| Aroclor-1254 | | | 250 | | |
| Aroclor-1260 | 50 | 100 | 250 | 500 | 750 |
| 2,4,5,6-tetrachloro-m-xylene | 4.0 | 8.0 | 20.0 | 40 | 80.0 |
| Decachlorobiphenyl | 4.0 | 8.0 | 20.0 | 40 | 80.0 |

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Calibration Stock Solutions, mg/L

| Compound | Mix | Conc. |
|---|---|---|
| alpha-BHC | AB | 5.0 |
| beta-BHC | AB | 5.0 |
| delta-BHC | AB | 5.0 |
| gamma-BHC (Lindane) | AB | 5.0 |
| alpha-Chlordane | AB | 5.0 |
| gamma-Chlordane | AB | 5.0 |
| Heptachlor | AB | 5.0 |
| Aldrin | AB | 5.0 |
| Heptachlor epoxide | AB | 5.0 |
| Endosulfan I | AB | 5.0 |
| Dieldrin | AB | 10.0 |
| 4,4'-DDE | AB | 10.0 |
| Endrin | AB | 10.0 |
| Endosulfan II | AB | 10.0 |
| 4,4'-DDD | AB | 10.0 |
| Endosulfan sulfate | AB | 10.0 |
| 4,4'-DDT | AB | 10.0 |
| Methoxychlor | AB | 50.0 |
| Endrin aldehyde | AB | 10.0 |
| Endrin ketone | AB | 10.0 |
| Chlordane – technical | TChlor | 1000 |
| Toxaphene | Tox | 500 |
| Aroclor-1016 | AR1660 | 100 |
| Aroclor-1221 | AR1221 | 100 |
| Aroclor-1232 | AR1232 | 100 |
| Aroclor-1242 | AR1242 | 100 |
| Aroclor-1248 | AR1248 | 100 |
| Aroclor-1254 | AR1254 | 100 |
| Aroclor-1260 | AR1660 | 100 |
| [1]Aroclor-1262 | AR1660 | 100 |
| [1]Aroclor-1268 | AR1660 | 100 |

[1] These Aroclors are required only for specified DoD projects.

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Breakdown Check Solution

| **Compound** | **Concentration µg/mL** |
|---|---|
| 4,4'-DDT | 100 |
| Endrin | 100 |

Surrogate Stock Solution

| **Compound** | **Concentration µg/mL** |
|---|---|
| 2,4,5,6-tetrachloro-m-xylene | 200 |
| Decachlorobiphenyl | 200 |

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## APPENDIX II

### Analysis Sequence-0.45mm ID

| Injection | Sample |
|---|---|
| | hexane rinse |
| 1 | breakdown check standard (PEM) |
| 2 | ICV standard INDABM |
| 3 | ICV standard multi-components(for PCB only analysis - can replace above 3 stds) |
| 4 - 13 | up to 10 subsequent sample or QC extracts |
| 14 | solvent rinse or IBLK (optional - not required) |
| 15 | CCV standard INDABM |
| 16 | CCV standard multi-components (for PCB only analysis - can replace above 2 stds) |
| 17 - 26 | up to 10 subsequent sample or QC extracts |
| Last | CCV standard INDABM |
| Last | CCV standard multi-components (for PCB only analysis - can replace above 2 stds) |

### Analysis Sequence-0.32mm ID

| Injection | Sample |
|---|---|
| | hexane rinse |
| 1 | breakdown check standard (PEM) |
| 2 | ICV standard INDABM |
| 3 | ICV standard multi-components(for PCB only analysis - can replace above 3 stds) |
| 4 - 13 | up to 10 subsequent sample or QC extracts or 12 hours |
| 14 | solvent rinse or IBLK (optional - not required) |
| 15 | CCV standard INDABM |
| 16 | CCV standard multi-components (for PCB only analysis - can replace above 2 stds) |
| 17 - 26 | up to 10 subsequent sample or QC extracts or 12 hours |
| last | CCV standard INDABM |
| last | CCV standard multi-components (for PCB only analysis - can replace above 2 stds) |

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## APPENDIX III

### Data Packet Order

I. QC SUMMARY

_____ Analysts 'Client' Comment (hard copy and floppy)
_____ Surrogate Recovery Summary Report
_____ Blank Spike Report
_____ MS/MSD Report
_____ Method Blank Summary

II. SAMPLE DATA:

_____ Organic Analysis Data Sheet
_____ Sample Confirmation Worksheet
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

III. STANDARD DATA:

_____ Linearity Report

Linearity Standards:
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

Second Source Standards:
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

Continuing Calibration Standards:
_____ CCV Report
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

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**APPENDIX III, continued**

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

_____ Method Blank
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

---

_____ Blank Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

---

_____ Matrix Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

---

_____ Matrix Spike Duplicate
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

---

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**APPENDIX III, continued**

V. Bench Sheets

_____ SDG Print out
_____ Total Solids, if applicable
_____ Extraction Bench Sheets
_____ Injection Sequence (ICAL Summary, Batch Summary)
_____ calculations, HTVR
_____ Standards Logs

VI. Reject Data:

DO NOT COPY DO NOT PAGINATE

Data not used to support sample results.
All data acquired but rejected due to out of control QC.
Non-routine standards used to support sample data should be placed at the last of the Standard Data section.

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## APPENDIX IV

**Method 8081B QA Requirements and Corrective Actions:**

| QA Element | Method Criterion | Laucks Criterion | Frequency | Corrective Action | Documentation |
|---|---|---|---|---|---|
| Initial Calibration | 5 calibration stds for linear, 6 stds for quadratic, for all single and multi-components, %RSD ≤15% for each analyte. | 5 calibration stds for linear, 6 stds for quadratic, for all single and multi-components, %RSD ≤15% for each analyte. | After performing major instrument maintenance or when persistent difficulties meeting CCV criteria occur. | Recalibrate or narrate faults if possible | Narrative |
| Second Source Standard | All analytes must be within 20%. | All analytes must be within 20%. | Performed after each initial calibration. | Re-analyze or obtain third source. | Kept on file with the ICAL. |
| Daily Calibration Check | Must be ±15% D for each analyte. | Must be ±15% D for each analyte. | At start of each new analytical sequence, or daily if RTs shift | Rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible) | Narrative |
| Continuing Calibration Verification | Must be ±15% D for each analyte. | Must be ±15% D for each analyte. | Every 10 samples or every 12 hours. | Rerun with new ICV, instrument maintenance, &/or recalibrate (or narrate if possible) | Narrative |
| Breakdown | Endrin and DDT breakdown must be <=15% | Endrin and DDT breakdown must be <=15% | At the beginning of each sequence. | Rerun, instrument maintenance | Narrative |
| Method Blank | Presence of any target analytes must be below MDL | Must be below ½ the reporting limit | Every analytical batch or 1 per 20 samples | Report quantitated contaminants with a "B" flag, or re-extract if necessary | Case narrative and corrective action form if re-extracted |

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| QA Element | Method Criterion | Laucks Criterion | Frequency | Corrective Action | Documentation |
|---|---|---|---|---|---|
| Surrogate Recovery | Limits to be determined by the lab. | Listed in LIMS | Every sample | Where contractually required, all surrogates must be within control limits. | Case narrative and corrective action form if re-extracted |
| Matrix Spike Recovery | Limits not specified. | Listed in LIMS | Every analytical batch or 1 per 20 samples | If other QC is in control, narrate; otherwise re-extract | Case narrative and corrective action form if re-extracted |
| MS/MSD RPD | Limits not specified | Listed in LIMS | Every analytical batch or 1 per 20 samples | If other QC is in control, narrate; otherwise re-extract | Case narrative and corrective action form if re-extracted |
| Blank Spike Recovery | Limits not specified. | Listed in LIMS | Every analytical batch or 1 per 20 samples | If other QC is in control, narrate; otherwise re-extract | Case narrative and corrective action form if re-extracted |
| Standard Reference Material (SRM) Recovery | Not required. | Not required. | Only if requested | Reanalyze or re-extract and reanalyze SRM | Case narrative and corrective action form if re-extracted |

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## APPENDIX V

**Compound Elution order on DB5 and DB608 Megabore (0.45 mm ID) Columns**

| DB5 | DB608 |
|---|---|
| Tetrachloro-m-xylene | Tetrachloro-m-xylene |
| alpha-BHC | alpha-BHC |
| beta-BHC | gamma-BHC (Lindane) |
| gamma-BHC (Lindane) | beta-BHC |
| delta-BHC | Heptachlor |
| Heptachlor | delta-BHC |
| Aldrin | Aldrin |
| Isodrin | Isodrin |
| Heptachlor epoxide | Heptachlor epoxide |
| gamma-Chlordane | gamma-Chlordane |
| Endosulfan I | alpha-Chlordane |
| alpha-Chlordane | Endosulfan I |
| 4,4'-DDE | 4,4'-DDE |
| Dieldrin | Dieldrin |
| Endrin | Endrin |
| Endosulfan II | 4,4'-DDD |
| 4,4'-DDD | Endosulfan II |
| Endrin aldehyde | 4,4'-DDT |
| Endosulfan sulfate | Endrin aldehyde |
| 4,4'-DDT | Endosulfan sulfate |
| Endrin ketone | Methoxychlor |
| Methoxychlor | Endrin ketone |
| Decachlorobiphenyl | Decachlorobiphenyl |

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## APPENDIX VI

**Compound Elution order on DBXLB and DB35ms Capillary (0.32 mm ID) Columns**

| DBXLB | DB35ms |
|---|---|
| Tetrachloro-m-xylene | Tetrachloro-m-xylene |
| alpha-BHC | alpha-BHC |
| gamma-BHC (Lindane) | gamma-BHC (Lindane) |
| beta-BHC | beta-BHC |
| delta-BHC | Heptachlor |
| Heptachlor | delta-BHC |
| Aldrin | Aldrin |
| Heptachlor epoxide | Heptachlor epoxide |
| gamma-Chlordane | gamma-Chlordane |
| alpha-Chlordane | alpha-Chlordane |
| Endosulfan I | Endosulfan I |
| 4,4'-DDE | 4,4'-DDE |
| Dieldrin | Dieldrin |
| Endrin | Endrin |
| 4,4'-DDD | 4,4'-DDD |
| Endosulfan II | Endosulfan II |
| Endrin aldehyde | 4,4'-DDT |
| 4,4'-DDT | Endrin aldehyde |
| Endosulfan sulfate | Endosulfan sulfate |
| Methoxychlor | Methoxychlor |
| Endrin ketone | Endrin ketone |
| Decachlorobiphenyl | Decachlorobiphenyl |

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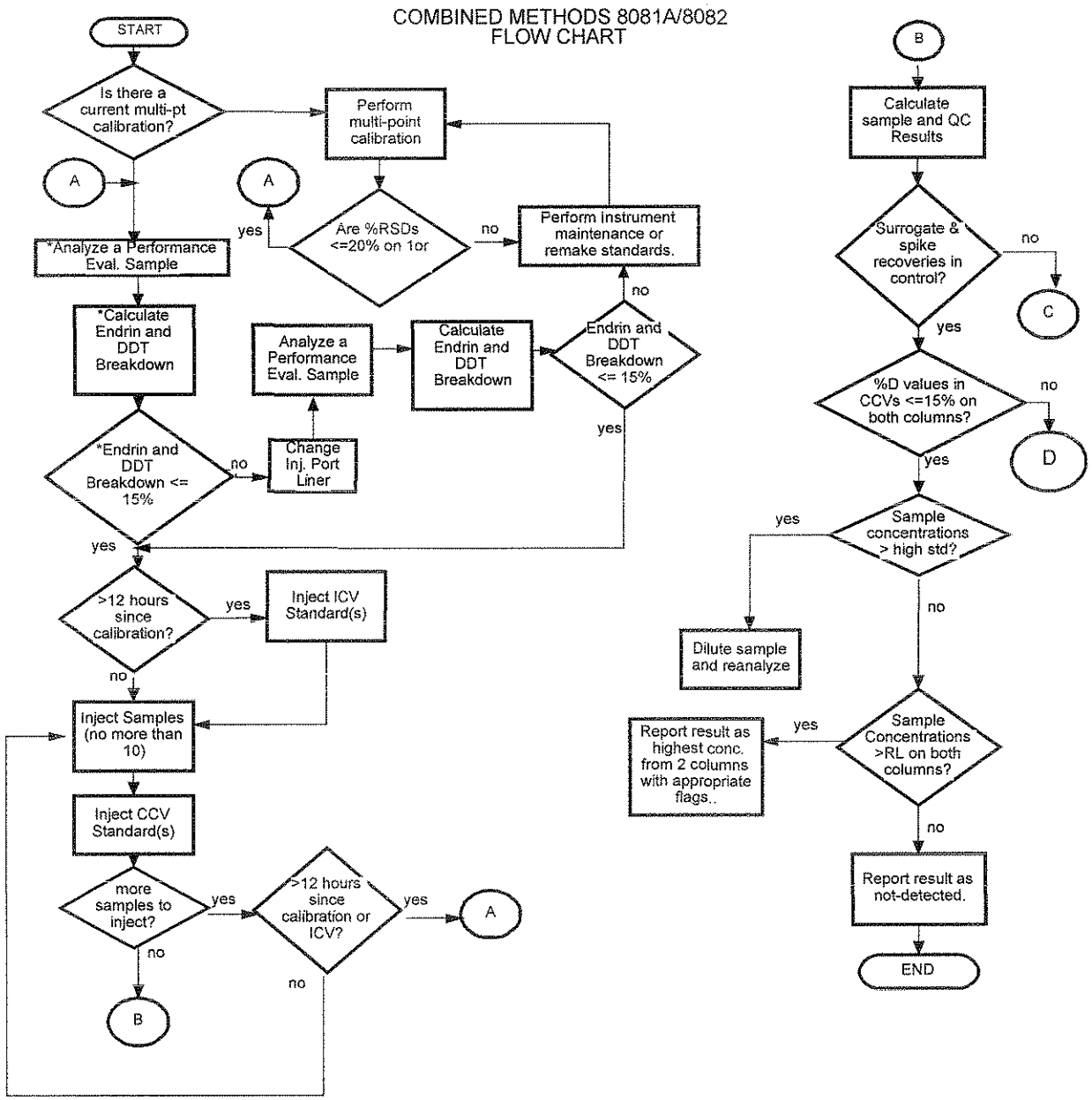
# APPENDIX VII

## Flow Chart




*These steps can be eliminated if analyzing for SW8082 compounds only

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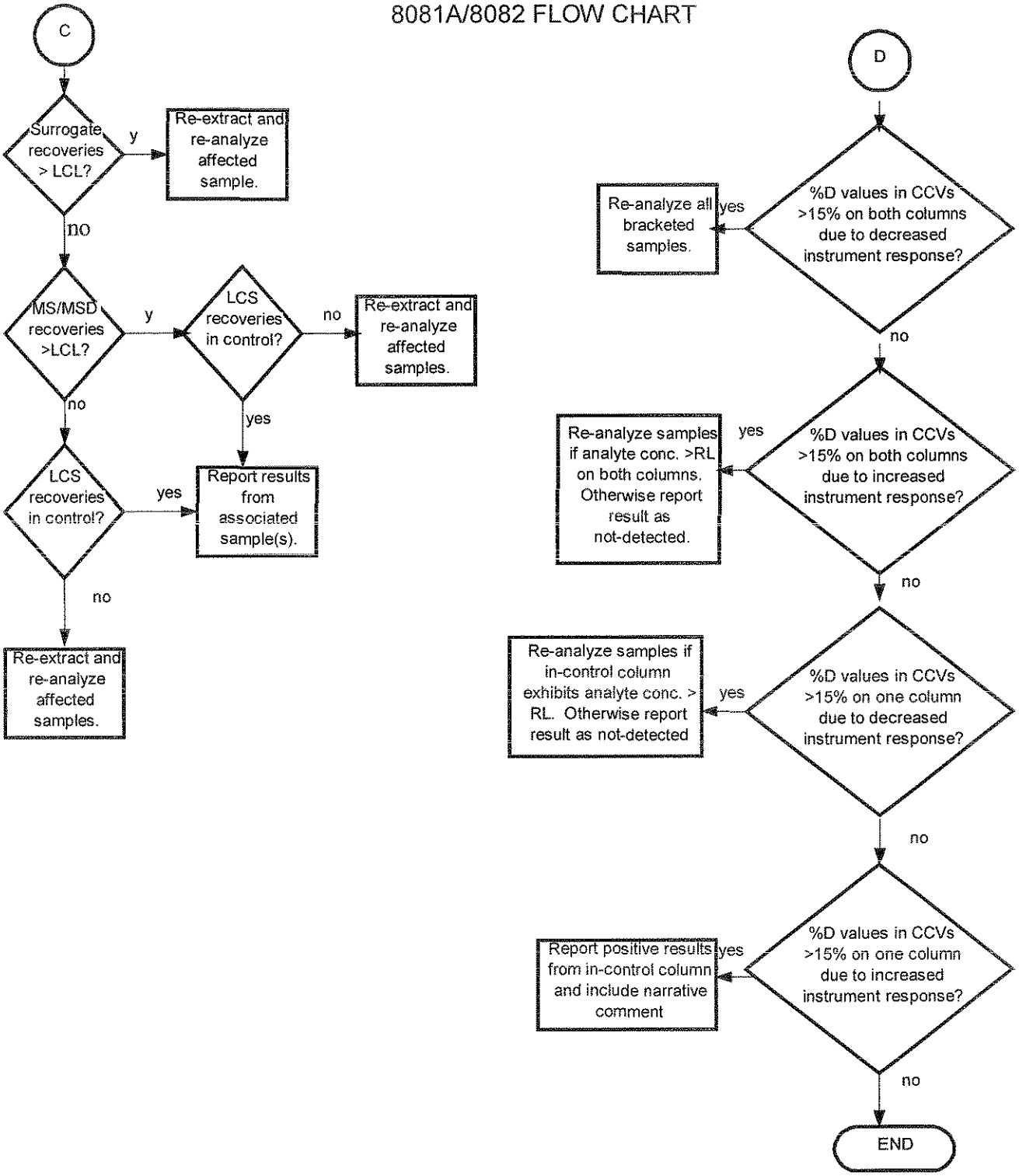
COMBINED
8081A/8082 FLOW CHART



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# Appendix VIII

## Agency Requirements for SW 8081

| QC Check | DoD | AFCEE | | SW 846 |
|---|---|---|---|---|
| | Acceptance Criteria | Acceptance Criteria | | Acceptance Criteria |
| **MDL Study** | See 40 CFR 136B. S/N > 3 for MDL verification. Every 12 months, or quarterly for MDL verification checks. | Detection limits ≤ 1/2 RL listed in appropriate AFCEE table. Every 12 months. | | |
| **Retention Time Window Width** | ± 3σ over a 72 hour period for each analyte | ± 3σ over a 72 hour period for each analyte | | ± 3σ over a 72 hour period for each analyte or ± 0.03 minutes, whichever is greater. |
| **Minimum Five Point Initial Calibration** | Option 1: RSD ≤ 20% | Option 1: Linear-RSD ≤ 20% for each analyte | | Option 1: Averaged - RSD ≤ 20% |
| | Option 2: Linear - r ≥ 0.995 | Option 2: Linear-least squares regression - r ≥ 0.995 | | Option 2: Linear - r ≥ 0.99 |
| | Option 3: Non-linear regression: coefficient of determination $r^2$ ≥ 0.99 | Option 3: Non-linear : coefficient of determination $r^2$ ≥ 0.99 | | Option 3: Non-linear - $r^2$ ≥ 0.99, minimum of 6 pts for 2nd order, 7pts for 3rd order AND %D ≤ 20% |
| **Second Source** | %D within ± 20% | Within ± 20% | | |

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| QC Check | DoD | AFCEE | | SW 846 |
|---|---|---|---|---|
| | Acceptance Criteria | Acceptance Criteria | | Acceptance Criteria |
| | | | | |
| **Retention Time** | Set at retention time of midpoint standard of ICAL | | | Set at retention time of first CCV of the analytical sequence, or of the midpoint standard if samples were ran after the ICAL |
| | | | | |
| **Retention time window position establishment for each analyte and surrogate** | Once per ICAL and at the beginning of the analytical shift. Position shall be set using the midpoint standard of the ICAL or the value in the CCV run at the beginning of the analytical shift. | | | |
| | | | | |
| **Retention time window verification for each analyte and surrogate** | Each caibration verification standard. Analyte within established window | | | |
| | | | | |
| **Continuing Calibration Verification** | Within ± 20%. | Within ± 15%. | | Within ± 15%. |
| | | | | |
| | Every 10 field samples, beginning and end of analytical | Every 10 samples, beginning and end of analytical sequence | | Beginning and end of each 12 hour analytical shift |

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| QC Check | DoD | AFCEE | | SW 846 |
|---|---|---|---|---|
| | Acceptance Criteria | Acceptance Criteria | | Acceptance Criteria |
| | | | | |
| | run | | | |
| | | | | |
| | | | | Required after every 20, recommended after 10 |
| | | | | |
| **Method Blank** | No analytes > 1/2 RL. No common laboratory contaminant analytes ≥ RL | No analytes ≥ RL | | Less than the laboratory detection limit OR Less than 5% of the regulatory limit OR Less than 5% of the sample result WHICHEVER IS GREATER |
| | | | | |
| **Laboratory Control Sample** | See appropriate DoD table, or use in-house limits if not listed | See appropriate AFCEE table | | Lab-derived |
| | | | | |
| **Matrix Spike** | Same as LCS | Same as LCS | | Lab-derived |
| | | | | |
| **Matrix Spike Duplicate** | RPD ≤ 30% | Water: RPD ≤ 30% | | Lab-derived |
| | | Soil: RPD ≤ 50% | | |
| | | | | |
| **Surrogate Spike** | Same as LCS | Same as LCS | | Lab-derived |
| | | | | |
| | | | | |
| | | | | |
| **Confirmation of positive results** | RPD ≤ 40, J flag if greater | RPD ≤ 40%, J flag if greater | | RPD ≤ 40% |

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| QC Check | DoD | AFCEE | | SW 846 |
|---|---|---|---|---|
| | Acceptance Criteria | Acceptance Criteria | | Acceptance Criteria |
| | | | | |
| | | | | |
| **Results reported between MDL and RL** | Apply J flags | Apply F flags | | |
| | | | | |
| **Allowable Sporadic ME/Failures** | 11 - 30 analytes – 1<br>1 – 10 analytes: 0 | | | |

# Appendix IX

## Control Limits*

Water

| Target Analyte | RL (µg/L) | MDL (µg/L) | Blank (µg/L) | MS/MSD | | | BS/BSD | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LCL | UCL | RPD | LCL | UCL | RPD |
| alpha-BHC | 0.05 | 0.0032 | 0.025 | 45 | 150 | 37 | 25 | 153 | |
| beta-BHC | 0.05 | 0.0042 | 0.025 | 41 | 166 | 32 | 60 | 144 | |
| delta-BHC | 0.05 | 0.004 | 0.025 | 54 | 175 | 35 | 38 | 174 | |
| gamma-BHC | 0.05 | 0.0037 | 0.025 | 35 | 142 | 27 | 43 | 163 | |
| Heptachlor | 0.05 | 0.0031 | 0.025 | 30 | 127 | 28 | 20 | 129 | |
| Aldrin | 0.05 | 0.0034 | 0.025 | 18 | 147 | 30 | 20 | 133 | |
| Heptachlor epoxide | 0.05 | 0.0035 | 0.025 | 41 | 149 | 27 | 39 | 162 | |
| Endosulfan I | 0.05 | 0.0036 | 0.025 | 37 | 171 | 9 | 53 | 146 | |
| Dieldrin | 0.1 | 0.007 | 0.05 | 39 | 143 | 27 | 55 | 133 | |
| 4,4'-DDE | 0.1 | 0.0086 | 0.05 | 57 | 161 | 11 | 48 | 150 | |
| Endrin | 0.1 | 0.0075 | 0.05 | 66 | 159 | 12 | 64 | 168 | |
| Endosulfan II | 0.1 | 0.0079 | 0.05 | 61 | 151 | 19 | 67 | 149 | |
| 4,4'-DDD | 0.1 | 0.0072 | 0.05 | 53 | 147 | 28 | 47 | 139 | |
| Endosulfan sulfate | 0.1 | 0.0064 | 0.05 | 48 | 153 | 37 | 49 | 154 | |
| 4,4'-DDT | 0.1 | 0.0088 | 0.05 | 51 | 149 | 14 | 46 | 134 | |
| Methoxychlor | 0.5 | 0.0349 | 0.025 | 54 | 149 | 28 | 52 | 135 | |
| Endrin aldehyde | 0.1 | 0.01 | 0.05 | 36 | 141 | 15 | 50 | 136 | |
| alpha-Chlordane | 0.05 | 0.0039 | 0.025 | 56 | 153 | 30 | 43 | 172 | |
| Endrin ketone | 0.1 | 0.0067 | 0.05 | 93 | 138 | 6 | 62 | 155 | |
| gamma-Chlordane | 0.05 | 0.0043 | 0.025 | 40 | 149 | 31 | 51 | 132 | |
| Chlordane | 1 | 0.021 | 0.5 | | | | | | |
| Toxaphene | 5 | 0.15 | 2.5 | | | | | | |
| Aroclor-1016 | 0.5 | 0.043 | 0.25 | 60 | 132 | 20 | 46 | 125 | |
| Aroclor-1221 | 0.5 | 0.039 | 0.25 | | | | | | |
| Aroclor-1232 | 0.5 | 0.084 | 0.25 | | | | | | |
| Aroclor-1242 | 0.5 | 0.051 | 0.25 | | | | | | |
| Aroclor-1248 | 0.5 | 0.086 | 0.25 | | | | | | |
| Aroclor-1254 | 0.5 | 0.16 | 0.25 | | | | | | |
| Aroclor-1260 | 0.5 | 0.045 | 0.25 | 59 | 139 | 21 | 50 | 143 | |

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Soil

| Target Analyte | RL (mg/Kg) | MDL (mg/Kg) | Blank (mg/Kg) | MS/MSD | | | BS/BSD | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | LCL | UCL | RPD | LCL | UCL | RPD |
| alpha-BHC | 1.7 | 0.3 | 0.85 | 46 | 141 | 34 | 53 | 136 | |
| beta-BHC | 1.7 | 0.38 | 0.85 | 43 | 158 | 30 | 59 | 146 | |
| delta-BHC | 1.7 | 0.34 | 0.85 | 48 | 162 | 37 | 66 | 156 | |
| gamma-BHC | 1.7 | 0.38 | 0.85 | 39 | 144 | 29 | 46 | 131 | |
| Heptachlor | 1.7 | 0.4 | 0.85 | 57 | 118 | 25 | 56 | 117 | |
| Aldrin | 1.7 | 0.38 | 0.85 | 47 | 139 | 28 | 59 | 126 | |
| Heptachlor epoxide | 1.7 | 0.4 | 0.85 | 36 | 140 | 52 | 50 | 133 | |
| Endosulfan I | 1.7 | 0.39 | 0.85 | 19 | 167 | 37 | 42 | 139 | |
| Dieldrin | 3.3 | 0.75 | 1.65 | 44 | 153 | 33 | 52 | 140 | |
| 4,4'-DDE | 3.3 | 0.83 | 1.65 | 20 | 190 | 47 | 57 | 153 | |
| Endrin | 3.3 | 0.86 | 1.65 | 55 | 167 | 34 | 48 | 154 | |
| Endosulfan II | 3.3 | 0.77 | 1.65 | 6 | 179 | 37 | 39 | 162 | |
| 4,4'-DDD | 3.3 | 0.74 | 1.65 | 49 | 170 | 41 | 29 | 160 | |
| Endosulfan sulfate | 3.3 | 0.74 | 1.65 | 41 | 155 | 34 | 41 | 150 | |
| 4,4'-DDT | 3.3 | 0.74 | 1.65 | 38 | 156 | 44 | 41 | 133 | |
| Methoxychlor | 17 | 3 | 8.5 | 67 | 142 | 39 | 61 | 118 | |
| Endrin aldehyde | 3.3 | 0.59 | 1.65 | 28 | 114 | 30 | 45 | 119 | |
| alpha-Chlordane | 1.7 | 0.44 | 0.85 | 19 | 174 | 48 | 38 | 189 | |
| Endrin ketone | 3.3 | 0.71 | 1.65 | 40 | 165 | 36 | 49 | 154 | |
| gamma-Chlordane | 1.7 | 0.37 | 0.85 | 26 | 150 | 49 | 53 | 132 | |
| Chlordane | 17 | 2.8 | 8.5 | | | | | | |
| Toxaphene | 170 | 6.4 | 85 | | | | | | |
| Aroclor-1016 | 17 | 3.1 | 8.25 | 42 | 128 | 16 | 62 | 130 | |
| Aroclor-1221 | 17 | 4.6 | 8.25 | | | | | | |
| Aroclor-1232 | 17 | 3.9 | 8.25 | | | | | | |
| Aroclor-1242 | 17 | 4.5 | 8.25 | | | | | | |
| Aroclor-1248 | 17 | 4.3 | 8.25 | | | | | | |
| Aroclor-1254 | 17 | 2.6 | 8.25 | | | | | | |
| Aroclor-1260 | 17 | 3.35 | 8.25 | 49 | 158 | 31 | 58 | 139 | |

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Water

| Surrogates | Limits | |
|---|---|---|
| | LCL | UCL |
| Tetrachloro-m-xylene | 10 | 129 |
| Decachlorobiphenyl | 32 | 149 |

Soil

| Surrogates | Limits | |
|---|---|---|
| | LCL | UCL |
| Tetrachloro-m-xylene | 61 | 129 |
| Decachlorobiphenyl | 75 | 158 |

* These control limits are laboratory-generated limits. Various agencies such as AFCEE and DoD require their own limits. The specific Agency control limits are located in the LIMS. Any DoD outlier must be qualified in accordance with Table B-2 in the DoD QSM. Any AFCEE outlier must be qualified in accordance with the current AFCEE QAPP.