Comprehensive Long-term Environmental Action Navy

CONTRACT NUMBER N62467-04-D-0055

N00164.AR.001071
NSWC CRANE
5090.3a

Resource Conservation and Recovery Act Addendum No. 1 to the Quality Assurance Project Plan for SWMUs 8, 15, 18, 19, 20 and The Old Gun Tub Storage Lot for Interim Measures at SWMU 7 (Old Rifle Range), SWMU 8 (Building 106 Pond), SWMU 13 (Mine Fill B) and SWMU 17 (PCB Burial/Pole Yard)

Naval Surface Warfare Center
Crane Division
Crane, Indiana

Contract Task Order 0020

November 2006

NAVFAC
Naval Facilities Engineering Command
Southeast
2155 Eagle Drive
North Charleston, South Carolina 29406

TETRA TECH NUS, Inc.

TETRA TECH NUS, INC.
661 Andersen Drive • Pittsburgh, PA 15220
Tel 412.921.7090 • Fax 412.921.4040 • www.tetratech.com

PITT-11-6-045

November 17, 2006

Project No. 112G00352

Mr. Tom Brent
NSWC Crane
Code 09510 Building 3245
300 Highway 361
Crane, Indiana 47522-5009

Reference: CLEAN Contract N62467-04-D-0055
Contract Task Order No. 0020

Subject: QAPP Addendum No. 1 for Naval Surface Warfare Center (NSWC) Crane Solid Waste Management Units (SWMUs) 8, 15, 18, 19, 20, and the Old Gun Tub Storage Lot for Interim Measures at NSWC Crane SWMUs 7, 8, 13, and 17

Dear Mr. Brent:

Remedial actions will be conducted at SWMUs 7 and 8. Five copies of the QAPP that will be used for confirmation sampling and waste characterization are enclosed. Comments on the draft QAPP, which was reviewed by USEPA Region 5 and IDEM, have been addressed as described in the September 6, 2006 and subsequent clarifications made in e-mails dated September 21, 2006 (Basinski to Gates) and September 26, 2006 (Basinski to Gates). IDEM approved the responses to these comments in an e-mail dated October 2, 2006 (Griffin to Brent).

Waste characterization information has been received from the SWMU 7 remedial contractor (D&T Environmental) and from the SWMU 8 remedial contractor (TolTest, Inc.). This information has been incorporated into Section 3 and Appendix A.

Please contact me at 412-921-8308 (e-mail: Ralph.Basinski@ttnus.com) or Valerie Plachy at 412-921-8389 (e-mail: (Valerie.Plachy@ttnus.com) regarding any questions or comments.

Sincerely,

Ralph R. Basinski
Task Order Manager

RRB/mlg
Enclosure

cc: Mr. Howard Hickey, NAVFAC-Midwest (letter and enclosure)
Ms. Debra Humbert, TtNUS, Inc. (letter only)
Mr. Mark Perry, TtNUS, Inc. (letter and unbound enclosure)
Ms. Valerie Plachy, TtNUS, Inc. (letter and enclosure)
Dr. Tom Johnston, TtNUS, Inc. (letter only)
Mr. David Hickey (letter and enclosure)
Mr. Cory Reiter (letter and enclosure)
Mr. Gary Resides (letter and enclosure)
Mr. Ralph Basinski, TtNUS, Inc. (letter and enclosure)
Project File – CTO 0020

REVISION 0
JULY 2006

RESOURCE CONSERVATION AND RECOVERY ACT ADDENDUM NO. 1 TO THE QUALITY ASSURANCE PROJECT PLAN FOR SWMUs 8, 15, 18, 19, 20, AND THE OLD GUN TUB STORAGE LOT FOR INTERIM MEASURES AT SWMU 7 (OLD RIFLE RANGE), SWMU 8 (BUILDING 106 POND), SWMU 13, (MINE FILL B), AND SWMU 17 (PCB BURIAL/POLE YARD)

NAVAL SURFACE WARFARE CENTER
CRANE DIVISION
CRANE, INDIANA

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

Submitted to:
Naval Facilities Engineering Command
Southeast
2155 Eagle Drive
North Charleston, South Carolina 29406

Submitted by:
Tetra Tech NUS, Inc.
661 Andersen Drive
Foster Plaza 7
Pittsburgh, Pennsylvania 15220

CONTRACT NUMBER N62467-04-D-0055
CONTRACT TASK ORDER 0020

JULY 2006

PREPARED UNDER THE SUPERVISION OF:

RALPH BASINSKI
TASK ORDER MANAGER
TETRA TECH NUS, INC.
PITTSBURGH, PENNSYLVANIA

APPROVED FOR SUBMITTAL BY:

RICHARD NINESTEEL, PE
QUALITY ASSURANCE MANAGER
TETRA TECH NUS, INC.
PITTSBURGH, PENNSYLVANIA

NAME TBD
PROJECT MANAGER
EMAC

ALLEN DEBUS
QUALITY ASSURANCE COORDINATOR
U.S. EPA REGION 5

HAK CHO
CORRECTIVE ACTION SECTION CHIEF
U.S. EPA REIGON 5

~~BILL GATES~~ Howard Hickey
REMEDIAL PROJECT MANAGER
NAVY SOUTHERN DIVISION
NAVAL FACILITIES ENGINEERING COMMAND SE

HARRY ROMBERG
LABORATORY QUALITY ASSURANCE OFFICER
LAUCKS TESTING LABORATORIES, INC.

DOUG GRIFFIN
CORRECTIVE ACTION PROJECT MANAGER
IDEM OFFICE OF LAND QUALITY

NAME TBD
QUALITY ASSURANCE MANAGER
EMAC

PETER RAMANAUSKAS
PERMITTING PROJECT MANAGER
U.S. EPA REGION 5

JOSE CISNEROS
WASTE MANAGEMENT BRANCH CHIEF
U.S. EPA REGION 5

TOM BRENT
SITE MANAGER
NSWC CRANE

HUGH PRENTICE
LABORATORY DIRECTOR
LAUCKS TESTING LABORATORIES, INC.

~~CRAIG RENDER~~
PROJECT CHEMIST
IDEM OFFICE OF LAND QUALITY

CORY REITER
PROJECT MANAGER - SWMU 7
D&T ENVIRONMENTAL, LLC

KAREN CAMPBELL
QUALITY ASSURANCE MANAGER
D&T ENVIRONMENTAL, LLC

GARY RESIDES
PROJECT MANAGER - SWMU 7
TOLTEST INC.

BILL WEBSTER
QUALITY ASSURANCE MANAGER
TOLTEST INC.

NAME TBD
PROJECT MANAGER - SWMU 13
SWMU 13 EMAC

NAME TBD
QUALITY ASSURANCE MANAGER
SWMU 13 EMAC

NAME TBD
PROJECT MANAGER - SWMU 17
SWVU 17 EMAC

NAME TBD
QUALITY ASSURANCE MANAGER
SWMU 17 EMAC

TABLE OF CONTENTS

SECTION **PAGE NO.**

1.0 PROJECT DESCRIPTION 1-1
1.1 DESCRIPTION OF QAPP ADDENDUM NO. 1 1-2
1.2 SCOPES OF WORK 1-2
1.2.1 SWMU 7, Old Pistol Range (OPR) at the Old Rifle Range (ORR) 1-2
1.2.2 SWMU 8, Building 106 Pond 1-3
1.2.3 SWMU 13 PCB Contamination Areas 1-5
1.2.4 SWMU 17, PCB Pole Yard 1-6
1.2 PROJECT TARGET PARAMETERS AND RATIONALE FOR SELECTION 1-7
1.2.1 Overall Analysis Summary 1-7

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES 2-1
2.1 MANAGEMENT 2-1
2.1.1 Indiana Department of Environmental Management Corrective Action Project Manager 2-2
2.1.2 U.S. EPA Oversight 2-2
2.1.3 Navy Project Manager 2-2
2.1.4 TtNUS Program and Project Management 2-2
2.1.5 EMAC Project Management 2-4
2.2 QUALITY ASSURANCE 2-6
2.2.1 Indiana Department of Environmental Management and U.S. EPA 2-6
2.2.2 TtNUS QA Manager 2-7
2.2.3 TtNUS Project QA Advisor 2-7
2.2.4 Laboratory Responsibilities 2-7
2.3 FIELD INVESTIGATION 2-9
2.3.1 Field Operations Leaders 2-10
2.3.2 Site QA/QC Advisor 2-11
2.3.3 Site Safety Officer 2-11
2.3.4 Technical Staff 2-11
2.4 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATIONS 2-12

3.0 FIELD OPERATIONS - SAMPLING AND ANALYSIS 3-1
3.1 GENERAL APPROACH 3-1
3.2 SAMPLE IDENTIFICATION SYSTEM 3-1
3.3 SAMPLE CUSTODY PROCEDURES 3-2
3.4 FIELD INSTRUMENT CALIBRATION PROCEDURES AND FREQUENCIES 3-2
3.5 SAMPLING LOCATIONS, ANALYSES, AND RATIONALES 3-3
3.5.1 General Sampling Requirements and Considerations 3-4
3.5.2 Field Instrument Calibration 3-5
3.5.3 SWMU 7 Sampling and Analyses 3-5
3.5.4 SWMU 8 Sampling and Analyses 3-9
3.5.5 SWMU 13 Sampling and Analysis 3-14
3.5.6 SWMU 17 Sampling and Analysis 3-16
3.6 QUALITY CONTROL REQUIREMENTS 3-19
3.7 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING 3-20
3.7.1 Confirmation Analysis Data 3-20
3.7.2 Waste Characterization Data 3-21
3.8 MANAGEMENT OF INVESTIGATION-DERIVED WASTE 3-21

TABLE OF CONTENTS

SECTION **PAGE NO.**

3.9 SAFETY 3-22
3.9.1 Health and Safety Plan 3-22
3.9.2 Health and Safety Training 3-22
3.9.3 Medical Approval and Personal Protective Clothing and Equipment 3-22
3.9.4 Safety Meetings 3-22
3.10 PERFORMANCE AND SYSTEM AUDITS 3-22
3.11 INSPECTION AND MAINTENANCE 3-23
3.12 FIELD CORRECTIVE ACTIONS 3-23
3.13 QUALITY ASSURANCE REPORTS TO MANAGEMENT 3-24

4.0 LABORATORY OPERATIONS AND DATA REDUCTION, REVIEW, AND MANAGEMENT 4-1
4.1 SAMPLE CUSTODY PROCEDURES 4-1
4.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES AND FREQUENCIES 4-1
4.3 LABORATORY ANALYTICAL AND MEASUREMENT PROCEDURES 4-1
4.3.1 Confirmation Sample Analyses 4-1
4.3.2 Waste Characterization Analyses 4-1
4.3.3 List of Project Target Compounds and Detection Limits 4-1
4.4 LABORATORY QUALITY CONTROL CHECKS 4-2
4.5 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING 4-2
4.6 LABORATORY PERFORMANCE AND SYSTEM AUDITS 4-2
4.6.1 Confirmation Analyses 4-2
4.6.2 Waste Characterization 4-2
4.7 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE 4-2
4.7.1 Confirmation Analysis 4-2
4.7.2 Waste Characterization 4-3
4.8 LABORATORY CORRECTIVE ACTION 4-3
4.8.1 Corrective Action Documentation 4-3
4.9.2 Corrective Action During Data Validation and Data Assessment 4-4
4.10 QUALITY ASSURANCE REPORTS TO MANAGEMENT 4-4
4.11 FINAL EVIDENCE FILES 4-5

5.0 REFERENCES 5-1

APPENDICES

A FIELD STANDARD OPERATING PROCEDURES
A.1 TETRA TECH NUS, INC. STANDARD OPERATING PROCEDURES
A.2 D&T ENVIRONMENTAL, LLC STANDARD OPERATING PRACTICES
A.3 TOLTEST INC. STANDARD OPERATING PROCEDURES

B LABORATORY STANDARD OPERATING PROCEDURES

TABLES

NUMBER

2-1 Key Project Personnel, SWMU 7 Interim Measures, Names, Phone Numbers, and Addresses
2-2 Key Project Personnel, SWMU 8 Interim Measures, Names, Phone Numbers, and Addresses
2-3 Key Project Personnel, SWMU 13 Interim Measures, Names, Phone Numbers, and Addresses
2-4 Key Project Personnel, SWMU 17 Interim Measures, Names, Phone Numbers, and Addresses
3-1 Summary of Sample Analyses, Container Types and Volumes, Preservation Requirements, and Holding Times for Field Analysis and Laboratory Confirmation Analysis of Soils and Sediments
3-2 Summary of Sample Analyses, Container Types and Volumes, Preservation Requirements, and Holding Times for Field Analysis and Laboratory Analysis of Aqueous Samples
3-3 Verification Sample Locations for SWMU 7 Excavation Floor and Side Wall Samples
4-1 Non-Calibration QC Sample Usage Frequencies, Acceptance limits, and Corrective Actions

FIGURES

NUMBER

1-1 Old Pistol Range Location Map, SWMU 7 - Old Rifle Range
1-2 Excavation and Demolition Plan, SWMU 8 - Building 106 Pond
1-3 Excavation Areas, SWMU 13 - Mine Fill B
1-4 Excavation Areas, SWMU 17 - PCB Capacitor Burial/Pole Yard
2-1 Project Organization Chart, SWMU 7 Interim Measures
2-2 Project Organization Chart, SWMU 8 Interim Measures
2-3 Project Organization Chart, SWMU 13 Interim Measures
2-4 Project Organization Chart, SWMU 17 Interim Measures
3-1 SWMU 7 Support Facilities
3-2 SWMU 7 Excavation Verification Sample Locations Example
3-3 SWMU 7 Interim Measures Support Facility Composite Sampling Example
3-4 SWMU 8 Verification Sampling - Excavation Areas and Support Facilities
3-5 SWMU 8 Interim Measures Excavation Sidewall Verification Sample Locations
3-6 SWMU 8 Interim Measures Support Facility Composite Sampling Example
3-7 SWMU 13 Verification Sampling - Excavation Areas and Support Facilities
3-8 SWMU 13 Interim Measures Excavation Verification Sample Locations Example
3-9 SWMU 17 Verification Sampling - Excavation Areas and Support Facilities
3-10 SWMU 17 Surface Soil Excavation Area Verification Sample Locations
3-11 SWMU 17 Stream and Drainage Channel Verification Sample Locations
3-12 Field Task Modification Request Form

ACRONYMS

%R	Percent Recovery
°C	Degrees Celsius
CAAA	Crane Army Ammunition Activity
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CTO	Contract Task Order
DNAPL	Dense Nonaqueous Phase Liquid
DQI	Data Quality Indicator
DQO	Data Quality Objectives
EDD	Electronic Data Deliverable
EDE	Electronic Data Exchange
EI	Environmental Indicator
EMAC	Environmental Multi-Award Contractor
ESM	Environmental Site Manager
FOL	Field Operations Leader
FTMR	Field Task Modification Request
GC/MS	Gas Chromatography/Mass Spectrometry
HASP	Health and Safety Plan
HPLC	High Performance Liquid Chromatography
HSM	Health and Safety Manager
IDEM	Indiana Department of Environmental Management
IDL	Instrument Detection Limit
IDW	Investigation Derived Waste
IM	Interim Measure
IWTP	Industrial Wastewater Treatment Plant
MDL	Method Detection Limit
mL	Milliliters
MS	Matrix Spike
MSD	MS Duplicate
NAVFAC SE	Naval Facilities Engineering Command Southeast
NFESC	Naval Facilities Engineering Service Center
NSWC	Naval Surface Warfare Center
OGTSL	Old Gun Tub Storage Lot

OPR	Old Pistol Range
ORR	Old Rifle Range
OSHA	Occupational Safety and Health Administration
PCB	Polychlorinated biphenyls
PE	Performance Evaluation
PM	Project Manager
PPE	Personal Protective Equipment
ProgMan	Program Manager
PTA	Pyrotechnic Test Area
QA	Quality Assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	Quality Control
RCRA	Resource Conservation and Recovery Act
RCRIS	Resource Conservation and Recovery Act Information System
RFI	RCRA Facility Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
RPM	Remedial Project manager
SOP	Standard Operating procedureP
SOW	Statement of Work
SSO	Site Safety Officer
SWMU	Solid Waste Management Unit
TCE	Trichloroethene
TOM	Task Order Manager
TtNUS	Tetra Tech NUS, Inc.
TV	Threshold Value
U.S. EPA	United States Environmental Protection Agency
USACE	United States Army Corps of Engineers
VOCs	Volatile Organic Chemicals
WP	Work Plan

1.0 PROJECT DESCRIPTION

The Naval Surface Warfare Center (NSWC) Crane prepared a Quality Assurance Project Plan (QAPP) [Tetra Tech NUS, Inc. (TtNUS), 2004] for a Phase III Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) at the following Solid Waste Management Units (SWMUs):

- SWMU 8, Building 106 Pond
- SWMU 15, Roads and Grounds Area

That QAPP also governed data collection for completion of the United States Environmental Protection Agency (U.S. EPA) Region 5 RCRA Conservation and Recovery Information System (RCRIS) code Environmental Indicators (EIs) Forms (Form CA725) for the following SWMUs:

- 18, Load and Fill Area Buildings
- 19. Pyrotechnic Test Area (PTA)
- 20, Crane Army Ammunition Activity (CAAA) Quality Assurance (QA)/Quality Control (QC) Test Area
- Old Gun Tub Storage Lot (OGTSL)

That QAPP was approved by U.S. EPA Region 5, and the required field work was conducted in accordance with the QAPP.

More recently, Interim Measures (IMs) Work Plans (IMWPs) were developed to govern IMs to be conducted at the following Crane SWMUs:

- Old Pistol Range (OPR) at SWMU 7, Old Rifle Range (ORR)
- SWMU 8, Building 106 Pond Area
- SMWU 13, Mine Fill B
- SWMU 17, PCB Pole Yard

The focus of the IMWPs is excavation of contaminated soil and sediment or other IMs that are designed to reduce or eliminate the extent and magnitude of contamination at each of these four sites.

The role of this QAPP Addendum No. 1 is explained in the next section.

1.1 DESCRIPTION OF QAPP ADDENDUM NO. 1

This QAPP Addendum No. 1 governs sampling and analyses for the purposes of remediation and waste disposal of materials removed from the four SWMUs listed above (7, 8, 13, and 17). Specifically, the following objectives will be achieved:

- Confirming that cleanup goals have been met.
- Provide data on residual contaminant concentrations in soils.

It is a supplement to the IMWPs (TtNUS, 2006a, 2006b, 2006c, and 2006d).

This QAPP Addendum No. 1 was prepared for the NSWC Crane facility, located in Crane, Indiana, through the Naval Facilities Engineering Command Southeast (NAFVAC SE) under Contract Task Order (CTO) 0020 for the Comprehensive Long-Term Environmental Action Navy (CLEAN) IV, Contract Number N62467-04-D-0055. For any work to be conducted under this QAPP Addendum No. 1 that is unique to this project, the appropriate information is presented in this document or references are provided to other documents containing the required information.

Site physical conditions are summarized in the IMWPs (TtNUS, 2006a; TtNUS, 2006b; TtNUS, 2006c; TtNUS, 2006d) for these four sites. Descriptions of the IMs to be conducted, except details of confirmation sampling and analyses and waste disposal sampling and analyses, are described in Section 3.0 of these four references. Additional details of the IMs are provided in the remaining sections of these four references. Therefore, only very brief descriptions of the sites are provided below.

1.2 SCOPES OF WORK

1.2.1 SWMU 7, OPR at the ORR

SWMU 7 consists of two separate areas, the ORR and the adjacent OPR. The IMWP for SWMU 7 was prepared for the OPR portion of SWMU 7. The OPR is approximately 10 acres in size and is located immediately adjacent to the northern end of the ORR. The OPR is immediately west of NSWC Crane Highway 8 and is bisected by unnamed intermittent tributaries to Turkey Creek. The ORR and OPR are inactive.

Historical operations conducted at the OPR consisted of small arms firing within two discrete areas (see Figure 1-1). The first area, referred to as Range 1, consists of an open field and an adjoining hillside. Reportedly, small arms shooters positioned themselves and fired westward toward metal-framed targets.

The hillside on the western side of one of the unnamed tributaries served as the backstop for the bullets. The second area, referred to as Range 2, is located immediately north of Range 1. Range 2 is also relatively flat and is currently overgrown with volunteer shrubs and native trees. The direction of firing was to the north, and a manmade earthen berm served as the backstop for the bullets.

The known nature and extent of contamination and the associated risks at the OPR site at SWMU 7 were established based on the results of investigations and studies conducted by the United States Army Corps of Engineers (USACE) and TtNUS. The results of these investigations identified two areas from which lead-contaminated soils are to be excavated, as described in the IMWP for SWMU 7 (TtNUS, 2006a). Soil will be excavated from the two berms used as backstops for bullets and lead shot fired during target practice.

Confirmation Sampling

Following soil excavation, samples will be collected from the soil remaining on the excavation floors and sidewalls to verify that residual lead concentrations in the exposed soils are acceptable (less than 400 mg/kg). Following completion of soil removal, TtNUS will also collect samples from beneath Environmental Multi-Award Contractor (EMAC) support facilities (decontamination pads, materials storage areas, and temporary access roads), if needed (samples to be analyzed for lead), to verify that surface soils below these facilities were not inadvertently contaminated during construction activities. Proposed excavation areas are identified on Figure 1-1. Details on the samples to be collected by TtNUS, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

Waste Characterization

In addition to the samples collected by TtNUS, the EMAC performing the excavations will collect waste characterization samples to satisfy the selected disposal facility permit requirements. Sampling requirements for waste characterization will be determined when the EMAC contractor selects a disposal facility. Details about the samples to be collected, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

1.2.2 SWMU 8, Building 106 Pond

SWMU 8 includes a surface pond located east and southeast of Buildings 106 and 107, respectively (Building 106 Pond). The Building 106 Pond occupies an area of approximately 2,550 square feet within the 5.8-acre area of SWMU 8. Currently the pond is surrounded by trees and a fence. The area east and northeast of the pond is wooded. South of the pond is an open grass area. In addition to these site

features, an electrical sub-station, overhead electrical lines, former industrial wastewater treatment facility, and monitoring wells are located within the Building 106 Pond site.

Historically the Building 106 Pond received process waters from Buildings 106 and 107. These buildings have historically been involved in the overhaul of projectile casings. Building 106 housed a cleaning process consisting of a caustic wash, a trichloroethene (TCE) degreaser, and a hydrochloric acid wash. Building 107 activities included refinishing of metal and wooden boxes. In the early 1970s, an industrial wastewater facility was constructed south of the Building 106 Pond, and the pond discharge was routed to this treatment facility. Following treatment in the industrial wastewater treatment facility, water was directed to the NSWC sanitary system. Prior to the construction of the industrial wastewater treatment facility, water was discharged from the pond through a scum rack to an unnamed drainage channel east of the pond.

The pond and the area immediately adjacent to the pond have been studied extensively, and the nature and extent of contamination and associated risks at the pond are based on the results of these investigations. As indicated in the IMWP for SWMU 8, the Building 106 Pond has been identified as the source of continuing contamination to the underlying groundwater (TtNUS, 2006b). Therefore, the IMWP for SWMU 8 requires excavation of the pond sediment and requires soils beneath the sediment to the depth of bedrock be excavated and transported to an off-site disposal facility. The former industrial wastewater treatment facility will be removed.

Confirmation Sampling

In addition to soil excavation, SWMU 8 IMWP activities include demolition of the former industrial wastewater treatment facility, dewatering of the SWMU 8 Building 106 Pond, treatment of the pond water, and discharge of the treated water to the NSWC Industrial Waste Treatment Plan (IWTP). Following soil excavation, TtNUS will sample any soil remaining on the excavation floors and sidewalls [samples to be analyzed for volatile organic compounds (VOCs)] to determine residual contamination levels. Following completion of the IMWP activities, samples will be collected from beneath EMAC contractor support facilities (decontamination pads, dewatering pad, water treatment system, materials storage areas, and temporary access roads), if needed (samples to be analyzed for VOCs), to verify that surface soils below these facilities were not inadvertently contaminated during remedial activities. A site layout map of the Building 106 Pond Area, including proposed excavation and demolition areas is provided as Figure 1-2. Details of the samples to be collected by TtNUS, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

Waste Characterization Sampling

In addition to the samples collected by TtNUS, the EMAC contractor will collect waste characterization samples to satisfy the selected disposal facility permit requirements (excavated soil samples) and to satisfy NSWC IWTP Requirements (treated water samples). Sampling requirements for excavated soil characterization sampling will be determined when the EMAC contractor selects a disposal facility. Sample requirements for the treated water samples will be determined by NSWC Crane. Details about the samples to be collected, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

1.2.3 SWMU 13 PCB Contamination Areas

PCBs were evidently released to soils in conjunction with use of Therminol boilers. These boilers heated a PCB heat transfer fluid that was used to melt explosives.

It is assumed that polychlorinated biphenyl (PCB) oils were heated in therminol boilers and then transferred to the SWMU 13 melt buildings (Buildings 166 and 171) where the oil was used as a heat-transfer medium in the melting of explosive mixtures. Some of the oil was evidently spilled in the vicinity of the boilers because PCB contamination was detected at relatively high concentrations in soils, primarily in shallow soils (TtNUS, 2005). Excavation will be conducted to remove PCB-contaminated soil, as described in the SWMU 13 IMWP (TtNUS, 2006c); sediments will be evaluated for the extent of contamination.

Confirmation Sampling

The excavation floors and sidewalls will be sampled (See Figure 1-3) and analyzed for PCBs to verify that the residual PCB concentrations in soil are acceptable. Acceptable concentrations are 25 mg/kg for soils deeper than 2 feet below ground surface (bgs) and 1 mg/kg for surface soils (i.e., soils within 2 feet of ground surface) and sediment in drainageways. Excavations will continue until acceptable concentrations of PCBs are achieved in the residual soils. Details about the samples to be collected by TtNUS, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

Waste Characterization Sampling

In addition to the samples collected by TtNUS, the EMAC contractor will collect waste characterization samples to satisfy the selected disposal facility requirements. Sampling requirements will be determined when the EMAC selects a disposal facility. Details about the samples to be collected, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

1.2.4 SWMU 17, PCB Pole Yard

SWMU 17 consists of three separate areas, the capacitor burial area, the pole storage yard, and the drainage ditch below the former transformer maintenance building (2721). SWMU 17 is approximately 17 acres in size and is located adjacent to Building 357. The area around Building 357 is currently active.

Historically, SWMU 17 has been used for 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, and storage of creosote-impregnated utility poles, some of which may have been contaminated with PCBs as a result of leaking transformers. It is known 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 the pole storage yard at SWMU 17 has been investigated extensively, and a previous IM to remove most of the contaminated soil was conducted in September 2004.

The nature and extent of contamination and the associated risks at SWMU 17 were determined based on the results of investigations and studies conducted by SAIC and TtNUS. The results of these investigations identified PCB contamination (Aroclor-1260) in surface soils and subsurface soils at SWMU 17. As indicated in the IMWP for SWMU 17, an IM was performed at SWMU 17 in 2004 by TolTest Inc. to remove the contaminated surface and subsurface soils associated with SWMU 17. However, verification samples collected following the excavation identified additional risk-causing PCB concentrations (Aroclor-1260) in surface soil and drainage channel sediments. Supplemental investigations performed by TtNUS (beginning in 2005) identified elevated PCB concentrations (Aroclor-1260) in several drainage channels and streams. As indicated in the IMWP, PCB-contaminated surface soils will be excavated and transported to an off-site disposal facility; sediments will be evaluated for the extent of contamination.

Confirmation Sampling

Following surface soil and sediment excavation, TtNUS will sample the soils and sediments remaining on the excavation floors and sidewalls to verify that the residual PCB concentrations in the exposed soils and sediments are acceptable. Following completion of soil removal, samples will also be collected from beneath EMAC contractor support facilities (decontamination pads, dewatering pad, materials storage areas, and temporary access trails), if needed (samples to be analyzed for PCBs), to verify that surface soils below these facilities were not inadvertently contaminated during construction activities. A site layout map of SWMU 17, including proposed excavation areas is provided as Figure 1-4. Details about

the samples to be collected, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

Waste Characterization Sampling

In addition to the samples collected by TtNUS, the EMAC contractor will collect soil characterization samples to satisfy the selected disposal facility permit requirements. Sampling requirements for soil characterization sampling will be determined when the EMAC contractor selects a disposal facility. Details about the samples to be collected, including descriptions of the sampling plan designs, are provided in Section 3.0 of this report.

1.2 PROJECT TARGET PARAMETERS AND RATIONALE FOR SELECTION

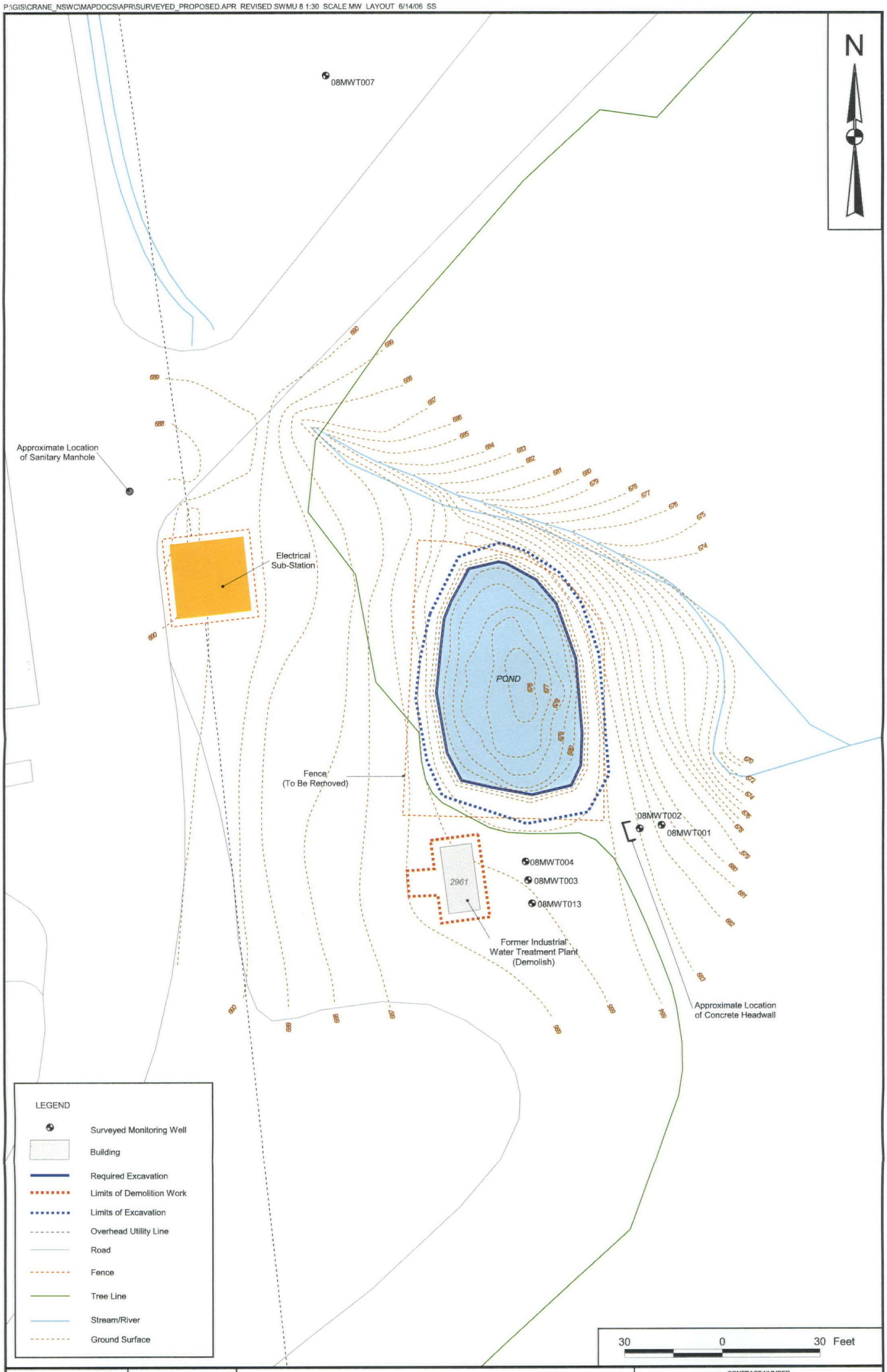
1.2.1 Overall Analysis Summary

Tables 3-1 and 3-2 present summaries of scheduled analyses and rationales for IM sampling. Specific compound lists corresponding to the analytical fractions listed in those tables are provided in Table 1-5 of the SWMU 8 and 15 QAPP (TtNUS, 2004).

P:\GIS\CRANE_NWS\MAPSOCS\APR\SWMU07.APR REVISED UPDATED OLD PISTOL RANGE LOCATION LAYOUT 7/11/06 SS

DRAWN BY S. STROZ	DATE 6/15/06	OLD PISTOL RANGE LOCATION MAP SWMU 7 - OLD RIFLE RANGE INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NUMBER CTO 0020
CHECKED BY T. JOHNSTON	DATE 7/11/06		APPROVED BY — DATE —
COST/SCHEDULE-AREA			APPROVED BY — DATE —
SCALE AS NOTED	NAVFAC		DRAWING NO. FIGURE 1 - 1 REV 0

P:\GIS\CRANE_NSWC\MAPDOCS\APR\SURVEYED_PROPOSED.APR REVISED SWMU 8 1:30 SCALE MW LAYOUT 6/14/06 SS



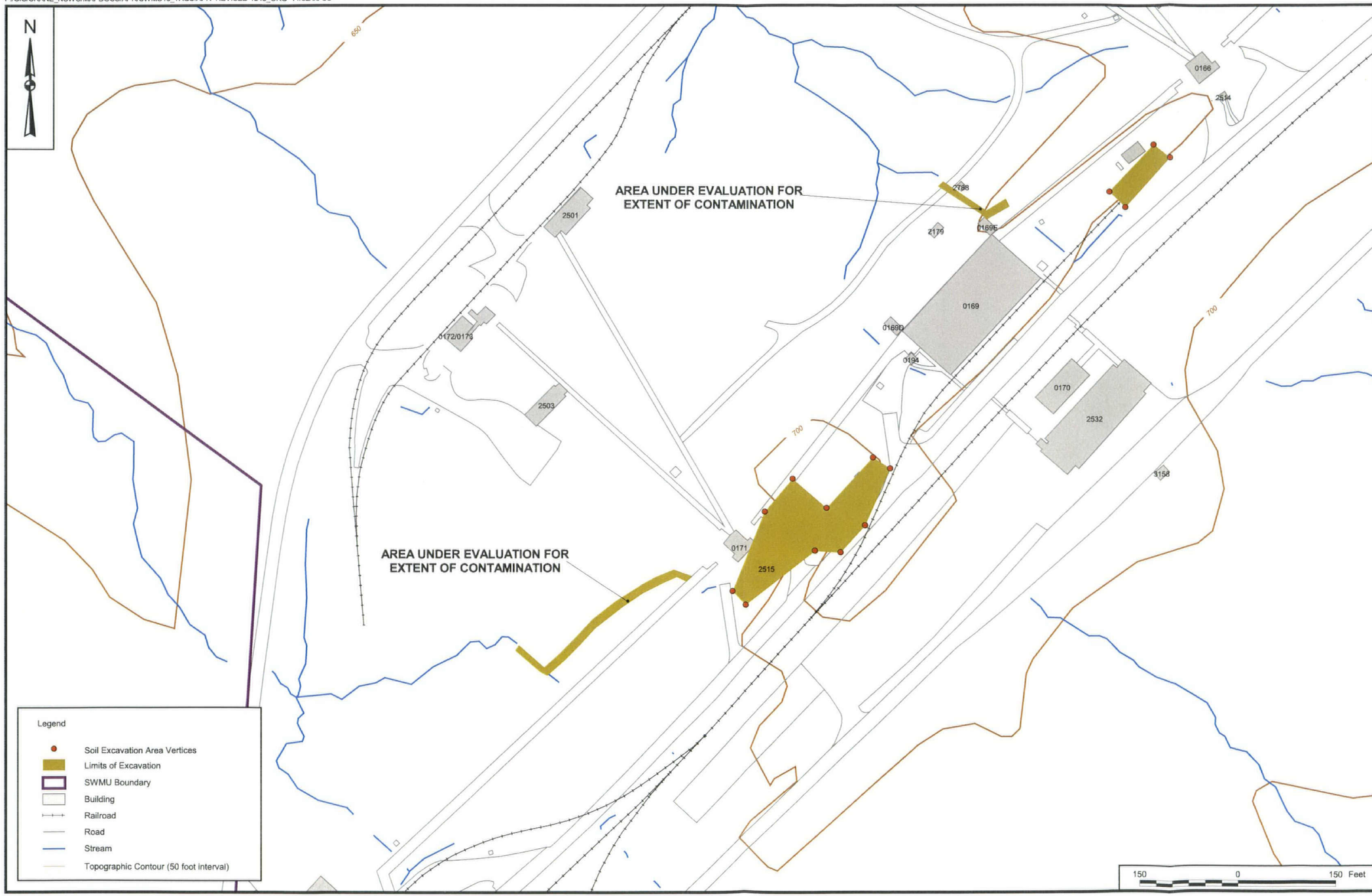
DRAWN BY	DATE
S. STROZ	6/14/06
CHECKED BY	DATE
T. SMITH	6/14/06
COST/SCHEDULE-AREA	
SCALE AS NOTED	

NAVFAC

EXCAVATION AND DEMOLITION PLAN
SWMU 8 - BUILDING 106 POND
INTERIM MEASURES QAPP ADDENDUM NO. 1
NSWC CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO 0020	
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
DRAWING NO. FIGURE 1 - 2	REV 0

P:/GIS/CRANE_NSWC/MAPDOCS/APR/SWMU13_TAGS.APR REVISED 1248_ORG 11/02/06 SS



CONTRACT NO.
112G00352

APPROVED BY ___ DATE ___

APPROVED BY ___ DATE ___

DRAWING NO. FIGURE 1 - 3

REV 0

EXCAVATION AREAS
SWMU 13 - MINE FILL B
INTERIM MEASURES QAPP ADDENDUM NO. 1
NSWC CRANE
CRANE, INDIANA

NAVFAC

DRAWN BY S. STROZ DATE 7/11/06

CHECKED BY V. PLACHY DATE 11/02/06

COST/SCHED-AREA

SCALE AS NOTED

P:\GIS\CRANE_NSWC\MAPDOCS\APR\SWMU17.APR REVISED UPDATED EXTENT OF INTERIM MEASURES EXCAVATION LAYOUT 11/02/06 SS

CONTRACT NO. 0020

APPROVED BY ___ DATE ___

APPROVED BY ___ DATE ___

DRAWING NO. FIGURE 1 - 4

REV 0

EXCAVATION AREAS
SWMU 17 - PCB CAPACITOR BURIAL/POLE YARD
INTERIM MEASURES QAPP ADDENDUM NO. 1
NSWC, CRANE
CRANE, INDIANA

NAVFAC

DRAWN BY S. STROZ DATE 6/15/06

CHECKED BY V. PLACHY DATE 11/02/06

COST/SCHED-AREA

SCALE AS NOTED

2.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

This section describes the project management, organization, and responsibilities of key personnel for IMs designed to remove contamination described in Section 1.0 at NSWC Crane SWMUs 7, 8, 13, and 17. Project organizational structure is the same for the four IMs, with flexibility to accommodate multiple remediation contractors (EMACs) and waste characterization laboratories.

TtNUS, on behalf of the United States Navy, is responsible for the preparation for this document and for oversight of IMs to ensure that they are completed satisfactorily at each of the four SWMUs. This will include confirmation sampling and analyses. The remedial contractor(s) are responsible for implementing the IMs for the contamination removal and associated waste disposal. This will include waste sampling and analyses.

The subcontracted laboratory for each SMWU is responsible for analyzing all confirmation samples in accordance with the analytical methods and additional requirements specified in this document. It also will be the responsibility of the analytical laboratory to properly dispose of unused sample aliquots.

2.1 MANAGEMENT

The roles and responsibilities described in this section are identified separately for TtNUS and the remediation contractors (EMACs). Because the roles and responsibilities of the EMACs are similar for each IM, the generic term EMAC is used when referring to those roles and responsibilities. Individual key persons responsible for work to be conducted under this QAPP Addendum No. 1 are identified in Figures 2-1 through 2-4 for SWMUs 7, 8, 13, and 17, respectively. Corresponding addresses and telephone numbers of key personnel are listed by organization in Tables 2-1 through 2-4. Each table of a given sequential number corresponds to the figure of the similar number (e.g., Table 2-3 corresponds to Figure 2-3). Indiana Department of Environmental Management (IDEM), U.S. EPA, Navy, TtNUS, and EMAC personnel specific to each IM are identified in the corresponding tables and figures.

TtNUS and EMACs will report directly to NAVFAC SE. TtNUS and the EMACs will maintain open channels of communication during field activities to facilitate resolution of problems as efficiently as possible. Personnel from the Navy will be actively involved and will coordinate with TtNUS and EMAC personnel as necessary. Responsibilities for program management, project management, field operations, and laboratory operations are discussed in the following sections, as appropriate. It is

intended that the individuals named will assume the designated responsibilities to the extent that the specific person is available to perform the stated activities.

2.1.1 Indiana Department of Environmental Management Corrective Action Project Manager

The IDEM Corrective Action Project Manager (PM), Mr. Doug Griffin, will oversee the implementation of the IMs at SWMUs 8 and 17. The IDEM PM represents IDEM's interests and will provide input from this perspective and lend general technical assistance to NSWC Crane field activities.

2.1.2 U.S. EPA Oversight

The U.S. EPA Region 5 PM, Peter Ramanauskas, will oversee the implementation of corrective measures at SWMUs 7 and 13. He will also represent U.S. EPA's interests and will provide input from this perspective and lend general historical and technical assistance to NSWC Crane field activities. U.S. EPA has delegated the RCRA program to IDEM.

2.1.3 Navy Project Manager

The Navy Remedial Project Manager (RPM), Mr. Bill Gates, acts as the focal representative for the United States Navy, providing management, technical direction, and oversight for all NSWC Crane project activities performed by contractors (e.g., TtNUS and EMACs) and their subcontractors. The Navy RPM will is responsible for ensuring that EMACs and their subcontractors receive a copy of this QAPP Addendum No. 1. In matters such as facilitation of site access, oversight, etc., the Navy RPM is assisted by the NSWC Environmental Site Manager (ESM), Mr. Tom Brent.

2.1.4 TtNUS Program and Project Management

TtNUS Program Manager

The TtNUS NAVFAC SE CLEAN Program Manager (ProgMan), Ms. Debra Humbert, provides operations, technical, and administrative leadership and oversees and supports quality policies. The ProgMan assigns project Task Order Managers (TOMs) and oversees their performance. The ProgMan also ensures the availability of technical and support resources for program operations, and maintains consistency in procedures and projects among CTO assignments. In these matters, the ProgMan is assisted by the TOM.

TtNUS Task Order Manager

The TtNUS TOM, Mr. Ralph Basinski, has overall responsibility for ensuring that the project meets U.S. EPA and IDEM objectives and Navy and TtNUS quality standards associated with IM oversight, sample collection, sample analyses, and data interpretation to determine whether IMs are completed satisfactorily. The TOM is responsible for the preparation and distribution of the QAPP, at the direction of the Navy RPM, to all parties (except EMACs and their subcontractors) connected with the project. The Navy RPM is responsible for distributing the QAPP Addendum to the EMACs and EMAC subcontractors. The TOM will report to the Navy RPM and is responsible for technical QC and project oversight. Additional responsibilities of the TOM are as follows:

- Ensuring timely resolution of project-related technical, quality, and safety questions associated with TtNUS operations.
- Functioning as the primary TtNUS interface with the Navy RPM, the NSWC Crane ESM, TtNUS field and office personnel, and laboratory and EMAC points-of-contact.
- Ensuring that TtNUS health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories.
- Monitoring and evaluating all TtNUS subcontractor performance.
- Coordinating and overseeing work performed by TtNUS field and office technical staff (including data validation, data interpretation, and report preparation).
- Coordinating and overseeing maintenance of all TtNUS project records.
- Coordinating and overseeing review of TtNUS project deliverables.
- Preparing and issuing final TtNUS deliverables to the Navy.
- Approving the implementation of corrective actions associated with the TtNUS portion of project quality.

TtNUS Project Chemist

The TtNUS Project Chemist, Mr. Edward Sedlmyer, has overall responsibility for ensuring that the project meets objectives from the standpoint of laboratory performance. The Project Chemist is responsible for the technical preparation of laboratory statements of work (SOWs) and work releases. Project Managers (PMs) of all subcontractor laboratories conducting confirmation sample analyses will report to the TtNUS Project Chemist. The Project Chemist will report to the TOM. Additional responsibilities of the Project Chemist are as follows:

- Providing technical advice to the TtNUS team on matters of project chemistry.
- Monitoring and evaluating subcontractor laboratory performance.
- Ensuring timely resolution of laboratory-related technical, quality, or other issues effecting project goals.
- Functioning as the primary interface with all subcontracted laboratories and the TOM.
- Coordinating and overseeing work performed by all subcontracted laboratories.
- Coordinating and overseeing review of laboratory deliverables.
- Recommending appropriate laboratory corrective actions.

TtNUS Health and Safety Manager

The TtNUS Health and Safety Manager (HSM), Mr. Matt Soltis, is responsible for the following:

- Providing technical advice to the TOM on matters of health and safety
- Overseeing the development and review of the Health and Safety Plan (HASP)
- Conducting health and safety audits
- Preparing health and safety reports for management

Each field event will have the support of a health and safety officer that will be assigned by the HSM.

2.1.5 EMAC Project Management

EMAC Project Managers

The EMAC PMs (see Figures 2-1 through 2-4 and Tables 2-1 through 2-4) have overall responsibility for ensuring that their SWMU-specific projects meet U.S. EPA and IDEM objectives and Navy and EMAC quality standards to ensure that IMs are completed satisfactorily. The EMAC PMs will report to the Navy

RPM and are responsible for technical QC and project oversight. Additional responsibilities of the EMAC PMs are as follows:

- Ensuring timely resolution of project-related technical, quality, and safety associated with EMAC operations.
- Functioning as the primary EMAC interface with the Navy RPM, the NSWC Crane ESM, TtNUS field personnel, and waste disposal facility points of contact.
- Ensuring that EMAC health and safety issues related to this project are communicated effectively to all personnel and off-site laboratories.
- Monitoring and evaluating all EMAC subcontractor performance.
- Coordinating and overseeing work performed by EMAC field staff (including excavation, waste segregation, sludge dewatering, and sampling to determine hazardous status of wastes prior to disposal).
- Coordinating and overseeing maintenance of all EMAC project records.
- Coordinating and overseeing review of EMAC project deliverables.
- Preparing and issuing final EMAC deliverables to the Navy.
- Approving the implementation of corrective actions associated with the SWMU-specific EMAC portion of project quality.

EMAC Field Operations Leaders

The EMAC Field Operations Leaders (FOLs), one for each IM, have overall responsibility for ensuring that the SWMU-specific IMs meet objectives from the standpoint of adequate removal of contaminated media and appropriate disposal of wastes. Each EMAC FOL will have autonomy with regard to deciding how best to implement the field portion of the IM, but completion of the IM will be determined by TtNUS on behalf of the Navy through confirmation sampling and analysis. The EMAC FOLs will report to the EMAC PMs. Additional responsibilities of the EMAC FOL are as follows:

- Providing technical advice to the field team on matters of remediation goals and implementation of IMWPs.

- Ensuring that wastes are sampled representatively to determine their status for waste disposal.

- Ensuring timely resolution of technical, quality, or other issues effecting project goals.

- Functioning as the primary interface with all SWMU-specific EMAC subcontractors and the EMAC PM.

- Recommending appropriate field corrective actions.

EMAC Health and Safety Managers

The EMAC HSMs are responsible for the following:

- Providing technical advice to the EMAC PMs on matters of health and safety.
- Overseeing the development and review of the HASP.
- Conducting health and safety audits.
- Preparing health and safety reports for management.

2.2 QUALITY ASSURANCE

This section identifies the QA responsibilities for the IMs. Responsibilities of IDEM, TtNUS, EMACs, and the analytical laboratories are discussed.

2.2.1 Indiana Department of Environmental Management and U.S. EPA

The IDEM chemist, Mr. Craig Pender, and U.S. EPA chemist, Mr. Allen Debus, have the responsibility to review and approve chemistry aspects of the QAPP Addendum No. 1 and to provide overall QA support and review. Mr. Pender is responsible for SWMUs 8 and 17: Mr. Debus is responsible for SWMUs 7 and 13. Additional responsibilities may include the following:

- Coordinating external performance and system audits of the contracted laboratories
- Reviewing and evaluating analytical field and laboratory procedures

2.2.2 TtNUS QA Manager

The TtNUS Quality Assurance Manager (QAM), Mr. Paul Frank, is responsible for overall QA for the TtNUS portion of the project. He reports directly to the TtNUS ProgMan. He acts on behalf of the United States Navy for project QA. The QAM is responsible for the following:

- Developing, maintaining, and monitoring QA policies and procedures.
- Providing training to TtNUS staff in QA/QC policies and procedures.
- Conducting systems and performance audits to monitor compliance with environmental regulations, contractual requirements, QAPP requirements, and corporate policies and procedures.
- Auditing project records.
- Monitoring subcontractor quality controls and records.
- Assisting in the development of corrective action plans and ensuring correction of nonconformances reported in internal or external audits.
- Ensuring that this QAPP Addendum meets TtNUS, Navy, and IDEM requirements.
- Overseeing the responsibilities of the TtNUS Project QA/QC Advisor.
- Preparing QA reports for management.

2.2.3 TtNUS Project QA Advisor

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

2.2.4 Laboratory Responsibilities

The subcontracted laboratory for each IM is responsible for analyzing all samples in accordance with the analytical methods and additional requirements specified in this QAPP Addendum No. 1. It will also be

the analytical laboratory's responsibility to properly dispose of unused sample aliquots. Responsibilities of key laboratory personnel are outlined in the following paragraphs.

Laboratory Project Managers

The Laboratory PMs will interface directly with the TtNUS Project Chemist, TtNUS TOM, and TtNUS QA Advisor and will perform the following tasks:

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

Laboratory Operations Manager

Responsibilities of the Laboratory Operations Manager include the following:

- Supporting the QA program within the laboratory
- Providing management overview of both production- and quality-related laboratory activities
- Maintaining adequate staffing to meet project analytical and quality objectives
- Approving all laboratory Standard Operating Procedures (SOPs) and QA documents
- Supervising in-house chain-of-custody documentation

Laboratory Quality Assurance Officer

The Laboratory QA Officer (QAO) will report directly to the Laboratory Operations Manager. The Laboratory QAO will be independent of laboratory production management to ensure that laboratory quality performance is assessed without schedule and cost considerations. Responsibilities of the Laboratory QAO include the following:

- Defining appropriate laboratory QA procedures and monitoring overall laboratory QA.

- Stopping work if a condition adverse to the quality of work is encountered, if QA or QC procedures are not followed, or if analytical out-of-control events are encountered that have not been corrected.

- Approving and maintaining document control of all QA documents and SOPs.

- Performing and/or implementing internal system and performance audits and verifying completion of corrective actions cited in audits.

- Directing laboratory participation in laboratory accreditation and certification programs.

Laboratory Sample Custodian

The Laboratory Sample Custodian will report to the Laboratory Operations Manager. Responsibilities of the Laboratory Sample Custodian include the following:

- Receiving and inspecting the incoming sample containers.
- Recording the conditions of the incoming sample containers.
- Signing appropriate documents.
- Verifying chain-of-custody records.
- Notifying the Laboratory PM of sample receipt and inspection.
- Assigning a unique identification number and customer number and entering each into the sample receiving log.
- With the help of the Laboratory PM, initiating transfer of the samples to appropriate lab sections.
- Controlling and monitoring access/storage of samples and extracts.

Laboratory Technical Staff

The laboratory technical staff will be responsible for sample analyses based on the analytical methods and requirements specified in this QAPP Addendum No. 1.

2.3 FIELD INVESTIGATION

TtNUS will be responsible for all field sampling and analysis activities related to confirming that the IMs are completed satisfactorily. The EMACs will be responsible for safely conducting the IMs and ensuring

that wastes are disposed properly. The TtNUS and EMAC field teams will be organized according to the activities planned. Field team members will be selected based on the type and extent of effort required. All team members will be appropriately skilled and trained for the tasks they are assigned to perform

2.3.1 Field Operations Leaders

The TtNUS and EMAC FOLs are responsible for coordinating all on-site personnel and for providing technical assistance, when required. The EMAC FOL, or designee, will coordinate and lead all excavations, dewatering, and waste removal operations. The EMAC FOL has authority to complete the field work in the most efficient manner while adhering to the requirements of this QAPP Addendum No. 1 and the HASP. The TtNUS FOL will conduct confirmation sampling activities. Each TtNUS and EMAC FOL will ensure the availability and maintenance of all sampling materials and equipment pertinent to their function. The TtNUS FOL is responsible for completing all sampling, field, and chain-of-custody documentation, will assume custody of all samples sent to the confirmation sampling laboratory, and will ensure the proper handling and shipping of samples. The TtNUS FOL will report directly to the TtNUS TOM but will have authority to guide field operations to efficiently conclude the scheduled work. Specific TtNUS and EMAC FOL responsibilities include the following:

- Ensuring that all health and safety requirements unique to these IMs are implemented.
- Functioning as the on-site communications link between field staff members, Site Safety Officer (SSO), the NSWC Crane ESM, and the TtNUS TOM or EMAC PM.
- Alerting off-site analytical laboratories of any special health and safety hazards associated with environmental samples.
- Overseeing the mobilization and demobilization of all field equipment and subcontractors.
- Coordinating and managing the field technical staff.
- Adhering to the work schedules provided by the TtNUS TOM and EMAC PM, as applicable.
- Ensuring the proper maintenance of site logbooks, field logbooks, and field recordkeeping.
- Initiating field task modification requests (field change orders) when necessary.

- Identifying and resolving problems in the field, resolving difficulties via consultation with the NSWC Crane ESM, implementing and documenting corrective action procedures, and providing communication between the field team and project management.

2.3.2 Site QA/QC Advisor

The FOLs (or designees) will act as the Site QA/QC Advisors and will be responsible for ensuring adherence to all QA/QC requirements as defined in this QAPP Addendum No. 1. Strict adherence to these procedures is critical to the collection of acceptable and representative data. The following is a summary of the Site QA/QC Advisor's responsibilities:

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

2.3.3 Site Safety Officer

The FOL (or designee) will also serve as the SSO. The duties of the TtNUS SSO are detailed in the TtNUS HASP; the duties of the EMAC SSO are detailed in the applicable EMAC HASP. The SSOs have stop-work authority, which can be executed upon the determination of an imminent safety hazard.

2.3.4 Technical Staff

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

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

2.4 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATIONS

All field personnel will have appropriate training to conduct the field activities to which they are assigned. Additionally, each site worker will be required to have completed a 40-hour course (and 8-hour refresher, if applicable) in Health and Safety Training as described under Occupational Safety and Health Administration (OSHA), 29 Code of Federal Regulations (CFR) 1910.120(b)(4).

Users of the X-ray fluorescence instrument must be properly trained to correctly operate the instrument by a certified user of the instrument or the instrument manufacturer.

TABLE 2-1

KEY PROJECT PERSONNEL
SWMU 7 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 2

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Peter Ramanauskas U.S. EPA Oversight U.S. EPA Region 5	EPA Region 5 77 West Jackson Blvd. Chicago, Illinois 60604	Phone: (312) 886-7890 FAX: (312) 353-4788
Howard Hickey Remedial Project Manager U.S. Navy NAVFAC ES	Department of the Navy Building 1A, Cod3e 45313 201 Decatur Avenue Great Lakes, IL 60080	Phone: (847) 688-5999 FAX: (847) 688-2319
Tom Brent Environmental Site Manager NSWC Crane	NSWC Crane Code RP3-TB B-3245 300 Highway 361 Crane, Indiana 47522-5009	Phone: (812) 854-6160 FAX: (812) 854-4177
Debbie Dills Officer in Charge of Construction NSWC Crane	NAVFAC MW PWD Crane Naval Support Activity Building 2516 Code PRCR22 300 Highway 361 Crane, Indiana 47522-5009	Phone (812) 854-3234 FAX: (812) 854-3800
Debra Humbert Program Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8968 FAX: (412) 921-4040
Richard Ninesteel Quality Assurance Coordinator Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8746 FAX: (412) 921-4040
Matt Soltis Health and Safety Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8912 FAX: (412) 921-4040
Ralph Basinski Task Order Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8308 FAX: (412) 921-4040
Dave Hickey Field Operations Leader Tetra Tech NUS	Tetra Tech NUS 800 Oak Ridge Turnpike, Suite 6000 Oak Ridge, TN 37830	Phone: (865) 220-4778 FAX: (865) 483-2014
Joseph Samchuck Data Validation Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8510 FAX: (412) 921-4040
Edward Sedlmyer Project Chemist Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8704 FAX: (412) 921-4040

TABLE 2-1

KEY PROJECT PERSONNEL
SWMU 7 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Tom Johnston Quality Assurance Advisor Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8615 FAX: (412) 921-4040
Ann Ho Project Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 957-2428 FAX: (206) 767-5063
Harry Romberg Lab QA Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 767-5060 FAX: (206) 767-5063
Drew Votaw Waste Characterization Laboratory Project Manager	Pace Analytical Services, Inc. 7726 Moller Road Indianapolis, IN 56268	Phone: (317) 875-5894 x105 FAX: (317) 872-6189
Cory Reiter Project Manager D&T Environmental, LLC	D&T Environmental 7008 Remington Ct. Edwardsville, IL 62025	Phone: (618) 659-4095 Cell: (314) 220-9369 FAX: (618) 659-4483
Todd Porter Health and Safety Officer D&T Environmental, LLC	D&T Environmental 7008 Remington Ct. Edwardsville, IL 62025	Ceel: (314) 220-9339
Karen Campbell Quality Assurance Manager D&T Environmental, LLC	D&T Environmental 7008 Remington Ct. Edwardsville, IL 62025	Phone: (317) 313-1136 FAX: (317) 867-4378

TABLE 2-2

KEY PROJECT PERSONNEL
SWMU 8 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 2

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Doug Griffin Corrective Action Project Manager Office of Land Quality Hazardous Waste Permits IDEM	Corrective Action Section Office of Land Quality Hazardous Waste Permits 100 N. Senate Avenue P. O. Box 6015 Indianapolis, Indiana 46206-6015	Phone: (313) 233-2710
Craig Pender Project Chemist Office of Land Quality IDEM	Office of Land Quality Hazardous Waste Permits 100 N. Senate Avenue P. O. Box 6015 Indianapolis, Indiana 46206-6015	Phone: (313) 234-0941
Howard Hickey Remedial Project Manager U.S. Navy NAVFAC ES	Department of the Navy Building 1A, Cod3e 45313 201 Decatur Avenue Great Lakes, IL 60080	Phone: (847) 688-5999 FAX: (847) 688-2319
Tom Brent Environmental Site Manager NSWC Crane	NSWC Crane Code RP3-TB B-3245 300 Highway 361 Crane, Indiana 47522-5009	Phone: (812) 854-6160 FAX: (812) 854-4177
Debbie Dills Officer in Charge of Construction NSWC Crane	NAVFAC MW PWD Crane Naval Support Activity Building 2516 Code PRCR22 300 Highway 361 Crane, Indiana 47522-5009	Phone (812) 854-3234 FAX: (812) 854-3800
Debra Humbert Program Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8968 FAX: (412) 921-4040
Richard Ninesteel Quality Assurance Coordinator Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8746 FAX: (412) 921-4040
Matt Soltis Health and Safety Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8912 FAX: (412) 921-4040
Ralph Basinski Task Order Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8308 FAX: (412) 921-4040
Dave Hickey Field Operations Leader Tetra Tech NUS	Tetra Tech NUS 800 Oak Ridge Turnpike, Suite 6000 Oak Ridge, TN 37830	Phone: (865) 220-4778 FAX: (865) 483-2014

TABLE 2-2

KEY PROJECT PERSONNEL
SWMU 8 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 2

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Joseph Samchuck Data Validation Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8510 FAX: (412) 921-4040
Edward Sedlmyer Project Chemist Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8704 FAX: (412) 921-4040
Tom Johnston Quality Assurance Advisor Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8615 FAX: (412) 921-4040
Ann Ho Project Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 957-2428 FAX: (206) 767-5063
Harry Romberg Lab QA Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 767-5060 FAX: (206) 767-5063
Nancy McDonalt Waste Characterization Laboratory Project Manager	STL Chicago 2417 Bond Street University Park, IL 60466	Phone: (708) 34-5200 FAX: (708) 534-5211
Gary Resides Project Manager TolTest Inc.	TolTest Inc. 5201 Jewell Lane Paducah, KY 42001	Phone: (270) 554-0880 Cell: (812) 709-1220
Bill Webster Health and Safety Officer TolTest Inc.	TolTest Inc. 508 W. Elnora Street Odon, IN 47526	Phone: (812) 636-8501 FAX: (812) 636-7572
Bill Webster Quality Assurance Manager TolTest Inc.	TolTest Inc. 508 W. Elnora Street Odon, IN 47526	Phone: (812) 636-8501 FAX: (812) 636-7572

TABLE 2-3

KEY PROJECT PERSONNEL
SWMU 13 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 2

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Peter Ramanauskas U.S. EPA Oversight U.S. EPA Region 5	EPA Region 5 77 West Jackson Blvd. Chicago, Illinois 60604	Phone: (312) 886-7890 FAX: (312) 353-4788
Bill Gates Remedial Project Manager U.S. Navy NAVFAC ES	Department of Navy NAVFAC SE Code OPGEVR 2155 Eagle Drive Charleston, South Carolina 29406	Phone: (843) 820-7360 FAX: (843) 820-7465
Tom Brent Environmental Site Manager NSWC Crane	NSWC Crane Code RP3-TB B-3245 300 Highway 361 Crane, Indiana 47522-5009	Phone: (812) 854-6160 FAX: (812) 854-4177
TBD Officer In Charge of Construction NSWC Crane	NSWC Crane Code **TBD** **TBD** 300 Highway 361 Crane, Indiana 47522-5009	Phone **TBD** FAX: **TBD**
Debra Humbert Program Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8968 FAX: (412) 921-4040
Richard Ninesteel Quality Assurance Coordinator Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8746 FAX: (412) 921-4040
Matt Soltis Health and Safety Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8912 FAX: (412) 921-4040
Ralph Basinski Task Order Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8308 FAX: (412) 921-4040
TBD Field Operations Leader Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: **TBD** FAX: **TBD**
Joseph Samchuck Data Validation Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8510 FAX: (412) 921-4040
Edward Sedlmyer Project Chemist Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8704 FAX: (412) 921-4040

TABLE 2-3

KEY PROJECT PERSONNEL
SWMU 13 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 2 OF 2

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Tom Johnston Quality Assurance Advisor Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8615 FAX: (412) 921-4040
Ann Ho Project Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 957-2428 FAX: (206) 767-5063
Harry Romberg Lab QA Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 767-5060 FAX: (206) 767-5063
TBD Waste Characterization Laboratory **Project Manager**	**TBD**	Phone: **TBD** FAX: **TBD**
TBD Project Manager EMAC **TBD**	**TBD**	Phone: **TBD** FAX: **TBD**
TBD Health and Safety Officer EMAC **TBD**	**TBD**	Phone: **TBD** FAX: **TBD**
TBD Quality Assurance Manager EMAC **TBD**	**TBD**	Phone: **TBD** FAX: **TBD**

EMAC = Environmental Multi-award Contractor
TBD = To be determined

TABLE 2-4

KEY PROJECT PERSONNEL
SWMU 17 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA
PAGE 1 OF 2

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Doug Griffin Corrective Action Project Manager Office of Land Quality Hazardous Waste Permits IDEM	Corrective Action Section Office of Land Quality Hazardous Waste Permits 100 N. Senate Avenue P. O. Box 6015 Indianapolis, Indiana 46206-6015	Phone: (317) 233-2710
Craig Pender Project Chemist Office of Land Quality IDEM	Office of Land Quality Hazardous Waste Permits 100 N. Senate Avenue P. O. Box 6015 Indianapolis, Indiana 46206-6015	Phone: (317) 234-0941 FAX: **TBD**
Bill Gates Remedial Project Manager U.S. Navy NAVFAC SE	Department of Navy NAVFAC SE Code OPGEVR 2155 Eagle Drive Charleston, South Carolina 29406	Phone: (843) 820-7360 FAX: (843) 820-7465
Tom Brent Environmental Site Manager NSWC Crane	NSWC Crane Code RP3-TB B-3245 300 Highway 361 Crane, Indiana 47522-5009	Phone: (812) 854-6160 FAX: (812) 854-4177
TBD Officer In Charge of Construction NSWC Crane	NSWC Crane Code **TBD** **TBD** 300 Highway 361 Crane, Indiana 47522-5009	Phone **TBD** FAX: **TBD**
Debra Humbert Program Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8968 FAX: (412) 921-4040
Richard Ninesteel Quality Assurance Coordinator Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8746 FAX: (412) 921-4040
Matt Soltis Health and Safety Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8912 FAX: (412) 921-4040
Ralph Basinski Task Order Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8308 FAX: (412) 921-4040
TBD Field Operations Leader Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: **TBD** FAX: **TBD**

TABLE 2-4

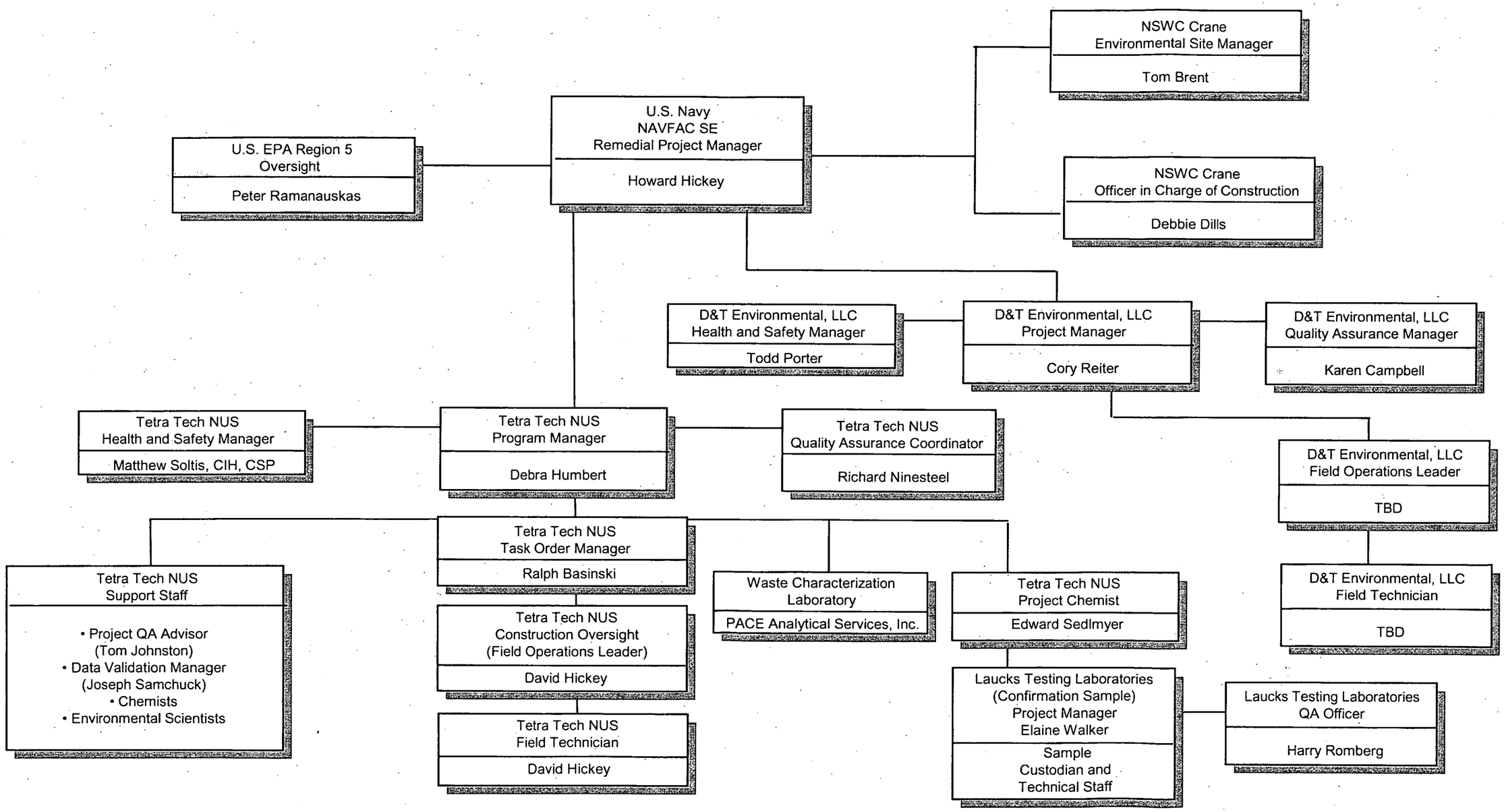
KEY PROJECT PERSONNEL
SWMU 17 INTERIM MEASURES
NAMES, PHONE NUMBERS, AND ADDRESSES
NSWC CRANE, CRANE, INDIANA

PERSON / TITLE / ORGANIZATION	ADDRESS	TELEPHONE
Joseph Samchuck Data Validation Manager Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8510 FAX: (412) 921-4040
Edward Sedlmyer Project Chemist Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8704 FAX: (412) 921-4040
Tom Johnston Quality Assurance Advisor Tetra Tech NUS	Tetra Tech NUS 661 Andersen Drive Pittsburgh, Pennsylvania 15220-2745	Phone: (412) 921-8615 FAX: (412) 921-4040
Ann Ho Project Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 957-2428 FAX: (206) 767-5063
Harry Romberg Lab QA Manager Laucks Testing Laboratories, Inc.	Laucks Testing Laboratories, Inc. 940 South Harney Street Seattle, WA 98108	Phone: (206) 767-5060 FAX: (206) 767-5063
TBD Project Manager EMAC **TBD**	**TBD**	Phone: **TBD** FAX: **TBD**
TBD Waste Characterization Laboratory **Project Manager**	**TBD**	Phone: **TBD** FAX: **TBD**
TBD Health and Safety Officer EMAC **TBD**	**TBD**	Phone: **TBD** FAX: **TBD**
TBD Quality Assurance Manager EMAC **TBD**	**TBD**	Phone: **TBD** FAX: **TBD**

EMAC = Environmental Multi-award Contractor
TBD = To be determined

FIGURE 2-1

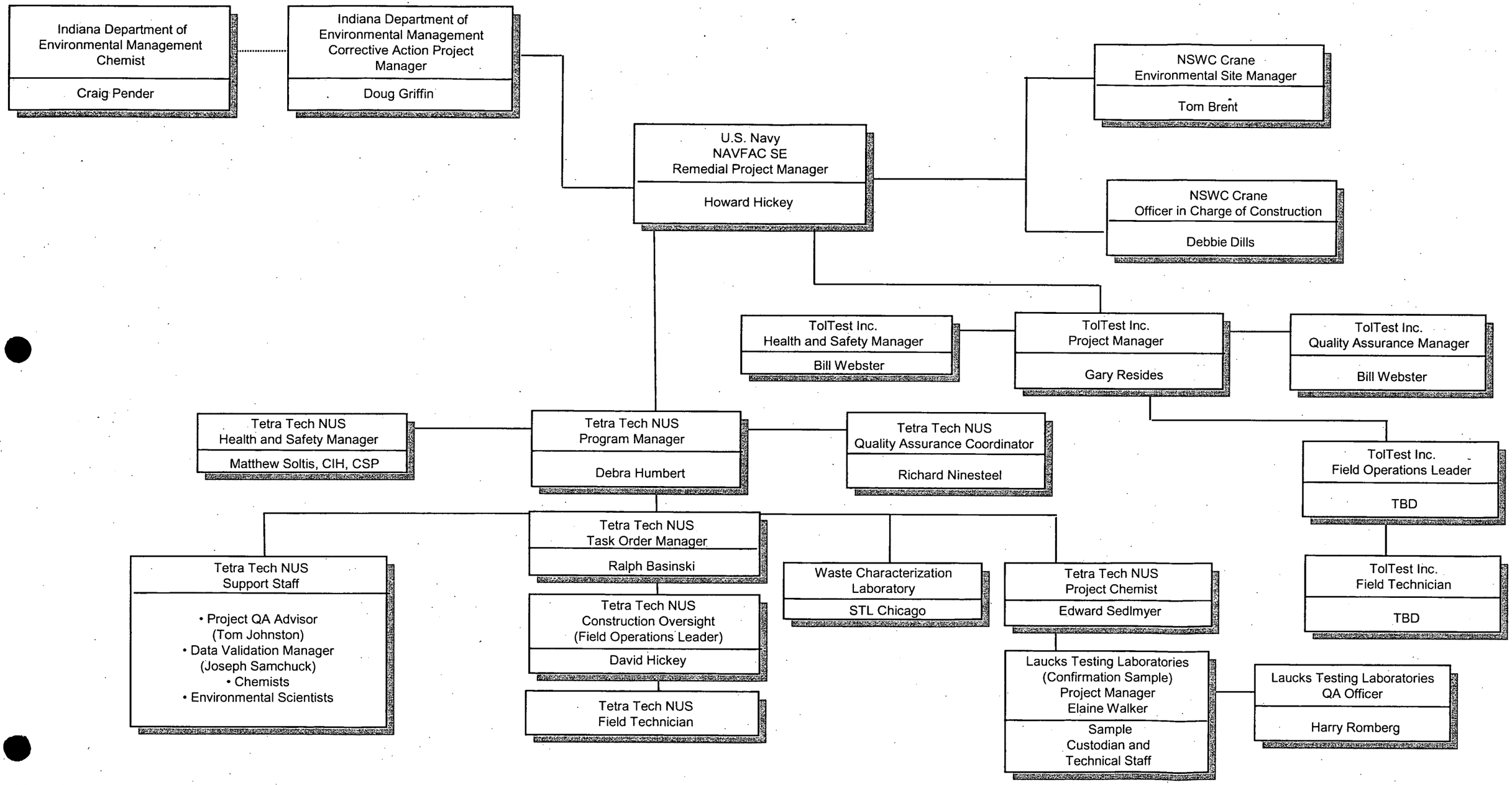
PROJECT ORGANIZATION CHART
SWMU 7 INTERIM MEASURES
NSWC CRANE, CRANE, INDIANA



TBD – To be determined

FIGURE 2-2

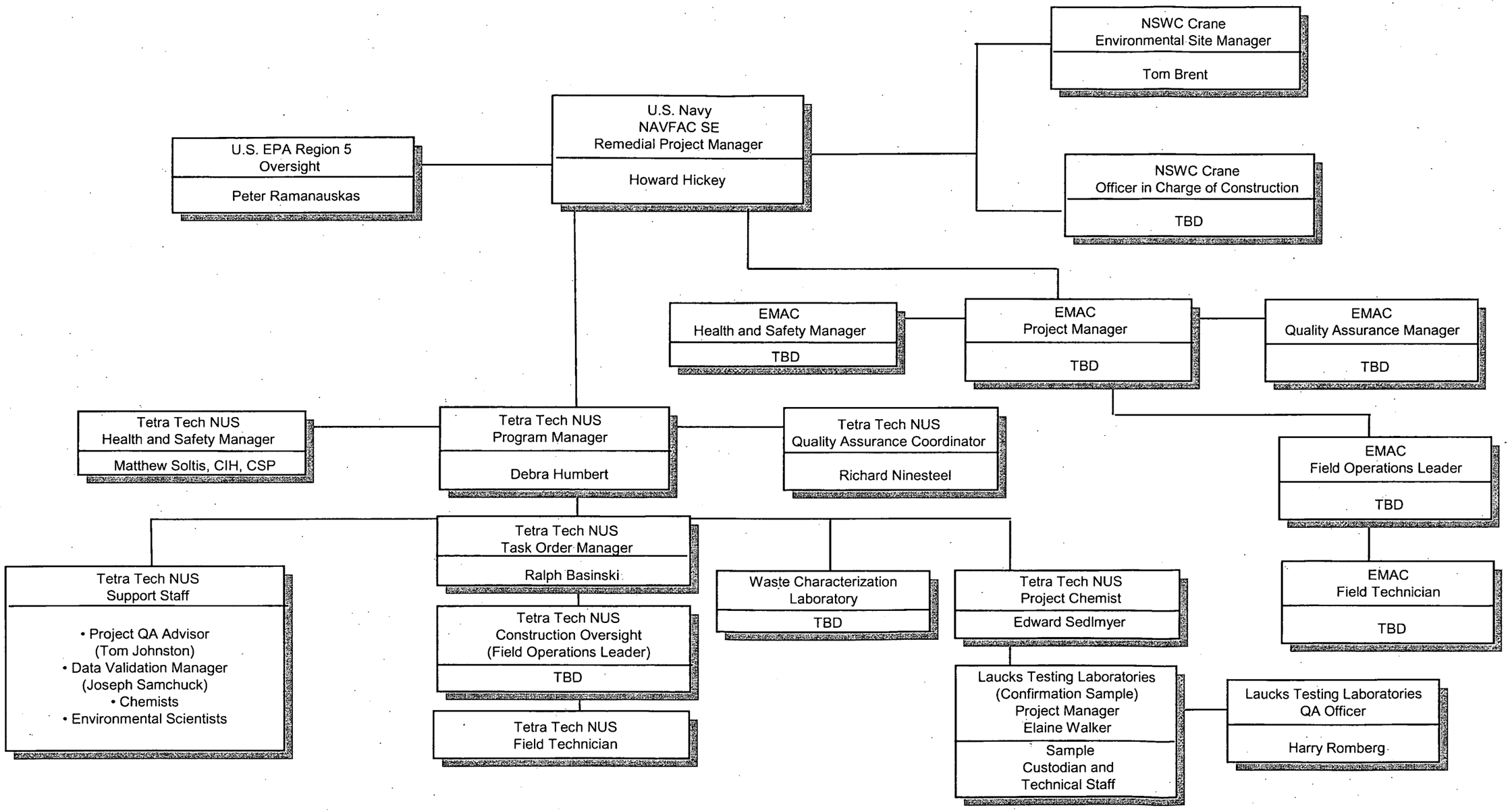
PROJECT ORGANIZATION CHART
SWMU 8 INTERIM MEASURES
NSWC CRANE, CRANE, INDIANA



TBD – To be determined

FIGURE 2-3

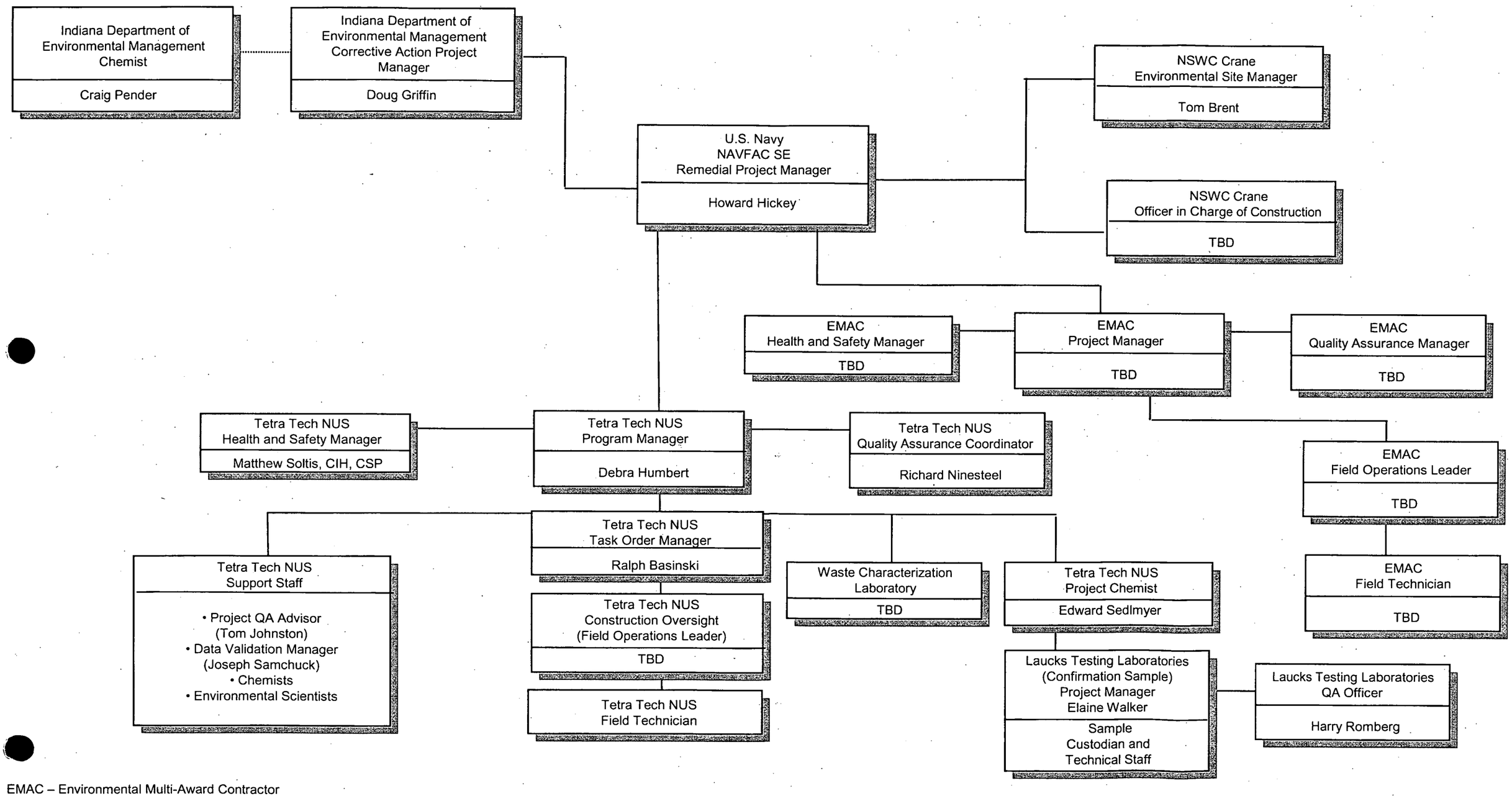
PROJECT ORGANIZATION CHART
SWMU 13 INTERIM MEASURES
NSWC CRANE, CRANE, INDIANA



EMAC – Environmental Multi-Award Contractor
TBD – To be determined

FIGURE 2-4

PROJECT ORGANIZATION CHART
SWMU 17 INTERIM MEASURES
NSWC CRANE, CRANE, INDIANA



EMAC – Environmental Multi-Award Contractor
TBD – To be determined

3.0 FIELD OPERATIONS - SAMPLING AND ANALYSIS

3.1 GENERAL APPROACH

This section describes sampling locations, methods, and rationales for the sampling activities to be conducted in support of the IMs to be conducted at SWMUs 7, 8, 13, and 17. Because this QAPP Addendum No. 1 is based on the existing SWMU 8 and 15 QAPP (TtNUS, 2004), the requirements regarding field operations as presented in TtNUS (2004) will be met during the four IMs, except if the information contained in this QAPP Addendum No. 1 conflicts with the existing QAPP. In those cases, this QAPP Addendum No. 1 shall take precedence over TtNUS (2004).

The equipment and procedures to be used for collecting, handling, preserving, recording, logging, and shipping samples to an analytical laboratory are presented in this QAPP Addendum No. 1. Corresponding SOPs can be found in Appendix A.1. Soil sampling will generally be performed using trowels and buckets.

Prior to any field activities, the TtNUS TOM and the EMAC PM will ensure that all field personnel read and understand this QAPP Addendum No. 1 and associated HASPs. TtNUS and the EMAC will each have organization-specific HASPs. Each worker or visitor to the site must sign an acknowledgment that he or she has read and understands the HASPs, as applicable.

The FOLs will ensure that all required field equipment for non-health-and-safety-related operations is available and operational, and the SSOs will ensure that all health-and-safety-related equipment is available and operational.

3.2 SAMPLE IDENTIFICATION SYSTEM

Confirmation Sampling

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 CTO020-01 (Appendix A.1). 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 name of the analytical laboratory to which the sample will be submitted.

Soil and sediment composite confirmation samples will be composited from multiple aliquots (discrete samples) collected to represent a specific area of an excavation floor or sidewall for all analyses except VOCs. Individual aliquots will be collected from 0 to 6 inches bgs for VOCs. For non-VOC analyses, composite samples will be formed by blending the aliquots together (blending is described in Section 3.5), where applicable, and the composite samples will be labeled in accordance with SOP CTO020-02 (Appendix A.1). Individual aliquots will be labeled as "A", "B", "C", or "D" to indicate sequential aliquots collected for compositing. The locations where the aliquots are acquired will be noted in the field log using a simple, hand-draw diagram. Individual soil and wastewater grab samples analyzed for VOCs will also be labeled in accordance with SOP CTO020-02 (Appendix A.1). Aqueous wastewater samples collected to determine disposal requirements will be individual grab samples.

The sampling time recorded on the chain-of-custody form, labels, and tags for duplicate samples will be 0000 so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory). Samples to be used as matrix spike (MS) and matrix spike duplicate (MSD) samples must be identified on the field documentation and chain-of-custody forms (see SOP CTO022-03 in Appendix A.1).

Waste Characterization Sampling

See Sections 3.5.3, 3.5.4, 3.5.5, and 3.5.6 for a detailed discussion.

3.3 SAMPLE CUSTODY PROCEDURES

Confirmation Sampling

The SOP governing sample custody, CTO0020-03 (Appendix A.1) of this QAPP Addendum No. 1.

Waste Characterization Sampling

See Sections 3.5.3, 3.5.4, 3.5.5, and 3.5.6 for a detailed discussion.

3.4 FIELD INSTRUMENT CALIBRATION PROCEDURES AND FREQUENCIES

Confirmation Sampling

Field instrument calibration requirements are specific to the instruments and chemical test kits. These requirements are governed by SOPs in Appendix A.1 or, in the case of PCB test kits, the calibration or

standardization instructions that accompany each test kit. It is important that the instructions in each PCB kit be followed accurately to ensure that the QC requirements specific to each kit are satisfied.

Waste Characterization Sampling

Field instruments are not expected to be used during waste characterization sampling.

3.5 SAMPLING LOCATIONS, ANALYSES, AND RATIONALES

Confirmation Sampling

This section presents sampling rationales, locations, QA/QC samples to be collected, and analyses to be performed for the sampling and analytical program. 24-hour turnaround times will be requested for all fixed-base laboratory analyses so as to minimize excavation equipment and field personnel down time while samples are analyzed. Field instruments and test kits will be used to guide the field activities, but fixed-base laboratory analyses will be considered the definitive data for determining whether IMs have been completed satisfactorily.

The overall rationale for confirmatory sampling is to ensure that remedial measures have been extensive enough that soil and sediments that are contaminated at concentrations above the media cleanup standard (MCS) have been removed from SWMUs 7, 13, and 17. In the case of SWMU 8, no MCS has been established for soils. The intent of confirmation sampling at SWMU 8 is to document residual concentrations in soils for the Navy to determine whether additional soils should be removed during this interim measure and also to provide information for use in the RFI and to determine whether further corrective measures are required. Confirmatory samples collected from excavations will be collected as surface soils at the exposed surface, but they will not represent surface soil depths when compared to the original ground elevations at the same locations. Limited depth information will be preserved in the sample numbers to convey to data users the depths bgs represented by the samples. However, continued excavation in a particular area may expose a yet deeper soil surface. The continued excavation at a particular location will be encoded in sample numbers (see SOP CTO020-02 in Appendix A.1), but it may be necessary to consult the field notes to estimate the depth below original ground elevation represented by any particular sample. This is not anticipated to be a problem because the goal of this project is to excavate until clean material is encountered, in which case the unacceptably contaminated material is assumed to have been removed.

If QC sampling and analysis is required by the off-site disposal facility, the facility's requirements for QC sample collection will be followed.

Tables 3-1 (solid samples) and 3-2 (aqueous samples) present summaries of the sample analyses, container types and volumes, preservation requirements, and holding times for the samples to be collected under this QAPP Addendum No. 1. The total number of samples to be collected is unknown; however, the samples to be collected are described below and in the IMWPs (TtNUS, 2006a through TtNUS, 2006d).

Waste Characterization Sampling

See Sections 3.5.3, 3.5.4, 3.5.5, and 3.5.6 for a detailed discussion.

3.5.1 General Sampling Requirements and Considerations

Confirmation Sampling

This section describes requirements and considerations that apply to sampling during IMs to be conducted at SWMUs 7, 8, 13, and 17.

Sample collectors have the option to use disposable equipment or reusable equipment for sampling. Disposable sampling equipment will not require decontamination. Reusable sampling equipment must be decontaminated between samples in accordance with SOP CTO020-04 (Appendix A.1). Equipment rinsate blanks and decontamination source water blanks, however, will not be collected because the chance of carryover from sample to sample (based on previous data from SWMUs 7, 8, 13, and 17) at the concentrations governing excavation is low. Furthermore, results from analysis of field blanks are not expected to influence the course of excavation. All sample collection must be recorded in accordance with the sampling procedures (SOPs CTO020-05 for soil in Appendix A.1 and Section 3.5.3 of this QAPP Addendum No. 1, "Waste Water Characterization," for aqueous samples).

Sample packaging and shipping for off-site laboratory analyses shall be conducted under chain of custody (see SOP CTO020-03 in Appendix A.1) in accordance with SOP CTO020-06 (Appendix A.1). One-day quick turnaround analyses shall be requested from the analytical laboratory. The request for quick turnaround analyses must be noted on each chain of custody.

Uniform random numbers were generated on the interval of 0 to 1 with Microsoft Excel 2003 (11.6560.6568) SP2 and converted to percentages for Table 3-3. This table indicates relative positions from where confirmation grab samples and composite samples will be collected in excavations. The use of Table 3-3 is described later for each SWMU. Example sample numbers are presented in the text

below to demonstrate how samples are to be numbered for the first samples to be collected for each environmental medium at each SWMU. See text below for SWMU-specific explanations and references to the tables.

Waste Characterization Sampling

See Sections 3.5.3, 3.5.4, 3.5.5, and 3.5.6 for a detailed discussion.

3.5.2 Field Instrument Calibration

Confirmation Sampling

Calibration checks of the field-portable XRF shall be conducted on each day of analyses in accordance with SOP CTO020-07 (Appendix A.1). The calibration checks shall occur at the beginning of analyses, every 4 hours thereafter or more frequently, and at the end of the analyses.

The Color-Tec apparatus will not be calibrated in the field because the color tubes are pre-calibrated by the manufacturer.

Calibration checks of the PCB test kits shall be conducted in accordance with the test kit instructions.

Waste Characterization Sampling

Field instruments are not expected to be used during waste characterization sampling.

3.5.3 SWMU 7 Sampling and Analyses

Confirmation Sampling and Analysis

The sizes and shapes of the excavations will be determined in the field, as described in Section 5.0 of the applicable IMWP (TtNUS, 2006a). Although the intent is to generally sample every 10-foot by 15-foot area of excavation floor (and corresponding excavation side walls in 1-foot depth intervals), the overall approach to collecting and analyzing composite soil samples is independent of the size and shape of the excavations. The goal of confirmation sampling will be to obtain samples that are representative of residual soils at the boundaries of the excavations. The sequence of sampling is described in the applicable IMWP (TtNUS, 2006a through 2006d). Figure 3-1 shows the areas to be excavated and hence where confirmation samples will be collected.

Table 3-3, Sets #1 and #2, present randomly selected coordinates for determining the locations where aliquots are to be collected to form composite samples in terms of the percentage of distance from one end of a rectangular excavation to the other end for both X- and Y-directions. The coordinates in Table 3-3 will be used as follows to identify the locations from which to obtain individual aliquots that will then be combined to form a composite sample:

1. If collecting aliquots from excavation floors or support facility, select Set #1.

2. If collecting aliquots from an excavation wall, select Set #2.

3. Select the next sequential sample number in the list of composite samples in the appropriate Table 3-3 set (e.g., the first sample selected for work conducted at the SWMU will be Sample 1).

Note 1: The sample numbers in Table 3-3 are essentially indices that help to identify sequential sets of coordinates, and they do not represent actual sample numbers.

4. Collect a soil or sediment aliquot from each of the four pairs (floors) or two pairs (side walls) of indicated X- and Y- coordinates for the selected composite sample.

Note 2: If the end of Table 3-3 is reached (e.g., Sample 20 for excavation floors), return to the beginning of the table and continue again to the bottom of the table in sequential order for each additional composite sample.

Note 3: Each aliquot should have the same volume.

Figure 3-2 shows a schematic diagram of how to measure the coordinates for each of the four (or two) sample aliquots that are to be composited from excavation floors (or side walls). Figure 3-3 (similar to Figure 3-2) shows a schematic diagram of how to measure the coordinates for each of the four sample aliquots that are to be composited from support facilities and the access road.

Excavated areas are not likely to be exactly rectangular. The sample collector will use professional judgment to measure the aliquot coordinates in the actual excavation while keeping as close as possible to the prescribed sampling scheme. After coordinates have been selected, the soil aliquots will be collected in accordance with SOP CTO020-05 (Appendix A.1). Individual aliquots for a particular composite sample will be labeled as "A," "B," "C," or "D" and shall be cross-referenced to the corresponding composite sample number on the Soil and Sediment Sampling Log Sheet. Each

composite sample must be labeled in accordance with SOP CTO0020-01 and CTO0020-02 in Appendix A.1). The first composite soil sample collected from an excavation floor will be labeled as follows (assuming it will be analyzed only for lead by field XRF): 07SOCF001A. The second composite soil sample to be collected (presumably from another sample grid location) and analyzed for lead by field XRF will be labeled as: 07SOCF002A. If the excavator returns to dig deeper at the first sampling grid location, the corresponding composite soil sample analyzed for lead by XRF would be 07SOCF001B. If excavation would continue at this same location to go yet deeper, the corresponding composite sample number will be: 07SOCF001C. Samples submitted for laboratory analysis that were splits of samples analyzed in the field will have the same number as the samples analyzed in the field.

After the aliquots are collected, they will be composited as follows:

1. Place the aliquots into a stainless steel bowl.

2. Mix soil or sediment aliquots of approximately equal volume (determined by visual observation) in the stainless steel bowl until the mixture is homogeneous in texture and color.

3. During mixing, inspect the mixture visually for evidence of lead shot, bullet fragments, and other debris associated with lead contamination. If lead shot, bullet fragments, or other SWMU 7-related contamination is visible, the Navy may decide to continue excavating without conducting any field or fixed-base laboratory analyses. Record the observation in field notes and consult the Navy.

4. Remove stones the size of a small pea or larger, sticks, root fragments, leaves, twigs, or other naturally occurring debris to ensure that the sample represents primarily soil.

The composite samples will be analyzed in accordance with SOP CTO020-07 (Field XRF Analysis of Soil and Sediment in Appendix A.1). As long as samples are being produced with field XRF lead concentrations greater than 400 mg/kg, 1 of 10 composite samples will be sent for laboratory verification of lead concentrations. If any sample at the limit of excavation has a lead concentration less than 400 mg/kg, the sample will be sent for laboratory verification. This will ensure that all final results supporting the conclusion to terminate excavation are verified with laboratory results. Each laboratory sample to be analyzed for lead shall be numbered in accordance with SOP CTO0020-02 (Appendix A.1). An example of how to number such a sample (assuming it was collected from the first composite sample collected from the excavation wall) is: 01SOCW001A. If excavation requires continued digging in that same location, the sample number, if submitted for laboratory analysis, will be: 01SOCLCW001B.

Soil Waste Characterization

The goal of waste characterization sampling is to characterize soils that will be disposed at an off-site disposal facility to determine whether the waste soils meet the facility's waste acceptance criteria. The remedial contractor (D&T Environmental, LLC) will utilize the following facility for disposal of waste material as a hazardous or non-hazardous waste:

S&S Grading of IL, Inc.
Rt. 3 Box 892
Sumner, IL 62466
(618) 936-9400

The hazardous designation will be determined based on whether the soil passes or fails the Toxicity Characteristic (TC) requirements for lead. The determination for passing or failing the TC requirements will be based on soil sampling from the Hillside Range 1 Berm and Range 2 Berm areas as described below.

Surface soil sampling will be accomplished by collecting a composite soil sample, from the three grid locations to be excavated from Range 2 Berm and the six grid locations to be excavated throughout from Hillside Range 1 Berm. Each grid sample will be collected from each of the areas designated for remediation in the SWMU 7 IMWP Figure 3-1 (TtNUS, 2006a). Each amount collected from each grid location will be of approximately equal volume and mass and will be collected in as unbiased a manner as practicable. This composite sample will be collected in accordance with SOP TNFLD006B (Appendix A.2). The sample shall be labeled and field records maintained in accordance with SOPs TNFLD010C and TNFLD002A (Appendix A.2).

The composite sample will be labeled and field records maintained in accordance with SOPs TNFLD010C and TNFLD002A (Appendix A.2). The soil quantities will be composited by mixing them in the field in accordance with SOP TNFLD006B (Appendix A.2) to obtain a uniform texture and color. A portion of this composite sample will be shipped to the following laboratory in accordance with SOP TNFLD010C (Appendix A.2):

Pace Analytical Services, Inc.
7726 Moller Road
Indianapolis, IN 46268
(317) 875-5894

The laboratory will leach the sample using the Toxicity Characteristic Leaching Procedure (TCLP) (SW-836 Method 1311) and will analyze the leachate for lead concentration using SW-846 Method 8260B.

If the reported lead concentration for the leachate is less than or equal to 5 milligrams per liter (mg/L), the soil will be shipped to a non-hazardous waste facility for disposal. If the reported leachate concentration is greater than 5 mg/L, the excavated soil will be shipped to a hazardous waste facility for disposal.

Laboratory results will be used to complete the waste profile form that will be provided to the disposal facility to document the classification of the waste as hazardous or non-hazardous.

3.5.4 SWMU 8 Sampling and Analyses

Soil and Sediment Confirmation Sampling and Analysis

The pond will be dewatered, sediment removed, any soil underlying the sediment removed to bedrock, and 1 foot of sidewall soil excavated to bedrock. Samples will be collected from the sidewall soils to provide information on residual VOC contamination. The Navy may elect to direct the EMAC to excavate additional sidewall soils based on the results of the analysis of the soils. Bedrock will not be sampled. Typical depth to bedrock is about 8 feet bgs but is expected to be deeper in some areas.

The intent of soil sampling along the pond wall is to represent every 20-foot interval around the pond perimeter with one grab sample that will be analyzed for VOCs. At least 10 samples will be collected for VOC analysis. VOCs are relatively mobile and volatile, so this relatively sparse sampling pattern is expected to be adequate for identifying areas that are unacceptably contaminated and subject to continued excavation. This will be verified with duplicate sample collection as described later. The goal will be to obtain samples that are representative of residual soils at the boundaries of the excavations. The sequence of sampling is described in the applicable IMWP (TtNUS, 2006b). Sampling will also occur under the support facilities to ensure that contamination has not been spread to those areas during remediation. Figure 3-4 shows the areas to be excavated and hence where confirmation samples will be collected. Notes on Figure 3-4 link the areas to be sampled to Figures 3-5 and 3-6. These latter figures are described later.

Table 3-3, Set #3, presents random coordinates for selecting grab samples in terms of the percentage of distance from one end of a rectangular excavation to the other end for both X- and Y-directions. The

coordinates in Table 3-3 will be used as follows to identify the locations from which to obtain each sample:

1. Select the next sequential sample number in the list of samples in Table 3-3 (the first sample selected for work conducted at the SWMU will be Sample 1). Ensure that coordinates from Set #3 are used.

Note 4: The sample numbers in Table 3-3 do not represent the sample numbers to be assigned to the composite samples. The Table 3-3 sample numbers should be viewed as index numbers used to distinguish one set of random coordinates from another.

2. Collect three aliquots of soil at the identified coordinates using Encore samplers in accordance with SOP CTO020-04 (Appendix A.1). Hold these three aliquots in a cooler of ice for shipment to fixed base laboratory. The fixed-base laboratory will extract and hold these samples pending instructions from the Navy.

3. Collect at least 50 grams of additional soil from the same location by scooping a portion of the sample into a glass jar. Cap the jar tightly, place it in a cooler of ice, and submit this sample for Color-Tec field analysis within 3 hours of collecting the sample. Color-Tec Analysis shall be conducted in accordance with SOP CTO020-08 (Appendix A.1). Label the Encores and glass jar with the same sample number in accordance with SOP CTO0020-2 (Appendix A.1).

Note 5: If the end of Table 3-3 Set #3 is reached for a given set of coordinates, return to the beginning of Set #3 in the table and continue to the end of the set in sequential order for each additional composite sample.

Figure 3-5 is a schematic diagram showing how to use the coordinates in Table 3-3, Set #3, to locate the place where each grab sample will be collected for VOC analyses along the pond banks. Similarly, Figure 3-6 shows how to use the coordinates to collect random samples from the IM support facilities and access road. The sample collector will use professional judgment to measure sampling coordinates in the excavation in a way that keeps as close as possible to the prescribed sampling scheme while accounting for the uneven surfaces of the areas to be sampled.

Record the total chlorinated VOC concentrations from the Color-Tec analyses in the field log.

Pond excavation will stop after a pre-defined volume of soil and sediment has been removed from the pond. At that point, the Navy will determine, based on the results of the Color-Tec analysis, whether additional excavation is required as described in Section 5.0 of the SWMU 8 IMWP (TtNUS, 2006b). If the Color-Tec analysis results are acceptable to the Navy and no further excavation is required, the fixed-base laboratory will be instructed to analyze the samples that represent where no further excavation is required. If further excavation is required, the fixed-base laboratory will be instructed to discard the previous samples and new samples will be collected at the boundary of the excavation for field and fixed-base laboratory analysis.

For every fifth pond sidewall sample collected, an additional sample will be randomly selected from the same 20-foot long section of pond bank using the coordinates in Table 3-3, Set #3. This sample will be considered a check sample whose results will be compared to the corresponding original sample to estimate the validity of collecting samples in a relatively sparse sampling pattern. It shall be labeled in accordance with the requirements of SOPs CTO0020-01 and CTO0020-02 in Appendix A.1. If the relative percent difference (RPD) of check sample field analyses exceed 70 percent, the sampling frequency will be increased to two randomly selected samples per 25-foot length of pond bank. RPDs shall be computed in accordance with Section 8.0 of the SWMU 8 and 15 QAPP (TtNUS, 2004) and shall be recorded in the field log.

Waste Sediment and Soil Characterization

The goal of waste characterization sampling is to characterize sediment that will be disposed at an off-site disposal facility to determine whether the waste sediments meet the facility's waste acceptance criteria. The remedial contractor (TolTest, Inc.) will utilize the following facilities for disposal of waste material as a hazardous or non-hazardous waste:

Non-Hazardous Waste Disposal:

Republic Services Sycamore Ridge Landfill
5621 E. Cottom Drive
Pimento, IN 47866
Phone: (317) 921-1667

Hazardous Waste Disposal:

Wayne Disposal, Inc.
49350 North I-94 Service Drive
Belleville, Michigan 48111
Phone: (800) 592-5489

Soils parameters and test methods are as listed in the following table:

Parameter	Analytical Method
SVOC	SW-846 8270C
TPH	SW-846 9071B
PCB	SW-846 8082
Total Metals	SW-846 6010B
Mercury	SW-846 7471
VOC	SW-846 8260B

This information will be used to complete the waste profile form and to determine whether the waste is toxicity characteristic hazardous.

The determination for passing or failing the TC requirements will be based on sampling from soils and sediments as described below.

At the time of waste sediment sampling, the sediment will have been dewatered and stockpiled so that sediment sampling points will be freely accessible and will not be covered with water. Therefore, TolTest, Inc. will accomplish both soil and sediment sampling by collecting the samples in accordance with TolTest Inc. SOP 8-3 (see Appendix A.3). The samples shall be labeled and field records maintained in accordance with TolTest Inc. SOPs 1-4, and 8-16 (Appendix A.3).

The sample shipments to the following laboratory for testing as listed previously listed will be in accordance with TolTest Inc. SOP 8-10 (Appendix A.3):

STL Chicago
2417 Bond Street
University Park, IL 60466
(708) 534-5200

Laboratory results will be used to complete the waste profile form that will be provided to the disposal facility to document the classification of the waste as hazardous or non-hazardous.

Wastewater Characterization

The goal of waste water characterization sampling is to characterize each tank of pre-treated wastewater accumulated from dewatering the pond to determine whether the wastewater meets the NSWC Crane criteria for transfer NSWC Crane Wastewater Treatment Plan (WWTP).

Once a tank has been filled, a single wastewater sample will be collected by TolTest personnel. It is assumed that mechanical mixing within the tank from the act of filling the tank will be sufficient to generate a homogeneous VOC concentration throughout the tank. If a dense non-aqueous phase liquid (DNAPL) is present, it will sink to the bottom of the tank, thus increasing DNAPL concentrations at the tank bottom. Collect a sample as follows:

1. Open the discharge valve (located at the bottom of the tank) and collect in a waste container at least three volumes of wastewater representing the volume of the sampling line between the tank and the exit point for the discharge. The estimated volume of the discharge line will be the line radius (not diameter) multiplied by itself, then multiplied by 3.14, then multiplied by the length of the line being sampled.

2. After purging this volume of wastewater, collect a sample by directly filling a 40-milliliter (mL) vial with minimal agitation or entrainment of air until there is a concave meniscus rising slightly above the top of the vial.

3. Place the Teflon®-line rubber septum, Teflon® side down, on the vial so as not to entrain any air, and screw the cap tightly over the septum.

4. Repeat this process two more times to yield three full VOC vials per sample, without air bubbles greater than ¼-inch diameter.

5. Immediately label the VOC vials in accordance with SOP 8-10 (Appendix A.3) and place them in a cooler with ice.

6. Return the excess purge water to the tank.

7. Submit the samples for 24-hour turnaround laboratory analysis for VOCs in accordance with Table 3-2 and SOP CTO020-03 (Appendix A.3).

Each wastewater characterization will be provided to the NSWC Crane WWTP personnel. For each tank accumulated, the NSWC WWTP personnel will determine whether the wastewater is suitable for transfer to the NSWC Crane WWTP surge tank.

3.5.5 SWMU 13 Sampling and Analysis

Soil Confirmation Sampling and Analysis

The sizes and shapes of the excavations at SWMU 13 will be determined in the field, as described in Section 5.0 of the applicable IMWP (TtNUS, 2006c). Although the intent is to sample every nominal 30-foot by 30-foot area of excavation floor and corresponding excavation side walls in 1-foot depth intervals, the overall approach to collecting and analyzing composite soil samples is independent of the size and shape of the excavations. The goal of confirmation sampling is to obtain samples that are representative of residual soils at the boundaries of the excavation. The sequence of sampling is described in the applicable IMWP (TtNUS, 2006c). Figure 3-7 shows the areas to be excavated and hence where confirmation samples will be collected. The drainage channel in the figure is currently being evaluated for extent of PCB contamination and remedial actions and the extent of remedial actions are not determined at this time.

Table 3-3, Sets #1 and #2, present random coordinates for composite soil sample aliquots in terms of the percentage of distance from one end of a rectangular excavation to the other end for both X- and Y-directions. The coordinates in Table 3-3 will be used as follows to identify the locations from which to obtain individual aliquots that will then be combined to form a composite sample:

1. If collecting aliquots from an excavation floor or support facility, select Set #1.

2. If collecting aliquots from an excavation wall or drainage channel bank, select Set #2.

3. Select the next sequential composite sample number in the list of composite samples in Table 3-3 (the first sample selected for work conducted at the SWMU will be Sample 1).

Note 6: The sample numbers in Table 3-3 do not represent the sample numbers to be assigned to the composite samples. The Table 3-3 sample numbers should be viewed as index numbers used to distinguish one set of random coordinates from another.

4. Collect a soil aliquot from each of the four pairs (floors and support facilities) or two pairs (side walls) of indicated X- and Y- coordinates for the selected composite sample.

Note 7: If the end of Table 3-3 is reached for a given set of coordinates (e.g., Sample 20 in Set #1 for excavation floors), return to the beginning of that set in the table and continue again to the end of the set in sequential order for each additional composite sample.

Figure 3-8 shows a schematic diagram of how to measure the coordinates for each of the four (or two) sample aliquots that are to be composited from excavation floors (or side walls).

Excavated areas are not likely to be exactly rectangular. The sample collector will use professional judgment to measure the aliquot coordinates in the actual excavation while keeping as close as possible to the prescribed sampling scheme. After coordinates have been selected, collect the soil aliquots in accordance with SOP CTO020-05 (Appendix A.1) and composite them as follows:

1. Mix soil aliquots of approximately equal volume (determined by visual observation) in a stainless steel bowl until the mixture is homogeneous in texture and color.

2. Remove stones the size of a small pea or larger, sticks, root fragments, leaves, twigs, or other naturally occurring debris to ensure that the sample represents primarily soil.

Analyze the composite samples in accordance with the instructions included in the PCB test kit identified in Table 3-1 and the instructions in SOP CTO0020-10 (Appendix A.1). Perform at least one calibration/standardization check daily. Record the total PCB concentration in the field log. QC checks for field analyses shall be conducted at the frequencies indicated in the test kit instructions. The information to be recorded includes but is not limited to the following:

- Analyst's name
- Date/time of analysis
- Test kit identifier, including serial number, if provided
- Reference to corresponding calibration/standardization check
- Sample number
- Measured concentration, including the measurement units

Report the total PCB concentration to the EMAC and the Navy RPM so a decision can be made concerning the need for continued excavation.

As long as samples are being produced with field PCB concentrations greater than 1 mg/kg in surface samples or 25 mg/kg in subsurface samples, send 1 of 10 composite samples will be sent for laboratory verification of PCB concentrations (as Aroclors). In this case, surface soil samples are samples collected within 2 feet of original grade as estimated through visual observation and simple field measurement with a tape measure or other suitable measuring device. If any sample at the limit of excavation has a total PCB concentration less than 1 mg/kg (surface soil) or 25 mg/kg (subsurface soil), that sample will be sent for laboratory verification of PCBs as Aroclors. This will ensure that a minimum of 10 percent or more of field analyses and all final results supporting the conclusion to terminate excavation are verified with laboratory results. For any sample that is analyzed in the field and the fixed-base laboratory, both the field and the laboratory analyzed portions will have the same sample number (see SOP CTP0020-02 in Appendix A.1).

Soil Waste Characterization

The goal of waste characterization sampling is to characterize soils that will be disposed at an off-site disposal facility to determine whether the waste soils meet the facility's waste acceptance criteria. **Details of soil waste characterization are being resolved by Navy and TtNUS and will be incorporated after they have been finalized.**

3.5.6 SWMU 17 Sampling and Analysis

Sediment Confirmation Sampling and Analysis

The sizes and shapes of the excavation areas and the soil and sediment volumes to be excavated will be determined in the field, as described in Section 5.0 of the applicable IMWP (TtNUS, 2006d). Although the intent is to sample 30-foot by 30-foot soil area and every 100-foot reach of drainage channel, the overall approach to collecting and analyzing composite soil or sediment samples is independent of the size and shape of the excavations. The goal will be to obtain samples that are representative of residual soils and sediments at the boundaries of the excavation. The sequence of sampling is described in the applicable IMWP (TtNUS, 2006d). Figure 3-9 shows the areas to be excavated and hence where confirmation samples will be collected.

Table 3-3, Sets #1 and #2, present random coordinates for composite sample aliquots in terms of the percentage of distance from one end of an excavation to the other end for both X- and Y-directions. The coordinates in Table 3-3 will be used as follows to identify the locations from which to obtain individual aliquots that will then be combined to form a composite sample:

1. If collecting aliquots from an excavation floor or support facility, select Set #1.

2. If collecting aliquots from an excavation wall or drainage channel bank, select Set #2.

3. Select the next sequential composite sample number in the list of composite samples in Table 3-3 (the first sample selected for work conducted at the SWMU will be Sample 1).

Note 8: The sample numbers in Table 3-3 do not represent the sample numbers to be assigned to the composite samples. The Table 3-3 sample numbers should be viewed as index numbers used to distinguish one set of random coordinates from another.

4. Collect a soil or sediment aliquot from each of the four pairs (floors and support facilities) or two pairs (side walls and banks) of indicated X- and Y- coordinates for the selected composite sample.

Note 9: If the end of Table 3-3 is reached for a given set of coordinates (e.g., Sample 20 in Set #1 for excavation floors), return to the beginning of that set in the table and continue again to the end of the set in sequential order for each additional composite sample.

Note 10: Each aliquot should have the same volume.

Figures 3-10 (excavation floors and side walls) and 3-11 (drainage channel beds and banks) show schematic diagrams of how to measure the coordinates for each of the four sample aliquots that are to be composited from excavation floors and drainage channel beds, and each of the two aliquots to be composited for excavation side walls and drainage channel banks.

Excavated areas are not likely to be exactly rectangular, especially those along stream channels. The sample collector will use professional judgment to measure the aliquot coordinates in the actual excavation while keeping as close as possible to the prescribed sampling scheme. Stream channel coordinates will be adjusted to follow the stream channel contour as though a rectangular grid was placed over the channel and allowed to conform to the channel surface. After coordinates have been selected, soil and sediment aliquots will be collected in accordance with SOP CTO020-05 (Appendix A.1) and composited as follows:

1. Mix soil or sediment aliquots of approximately equal volume (determined by visual observation) in a stainless steel bowl until the mixture is homogeneous in texture and color.

2. Remove stones the size of a small pea or larger, sticks, root fragments, leaves, twigs, or other naturally occurring debris to ensure that the sample represents primarily soil.

Analyze the composite samples in accordance with the instructions included in the PCB test kit identified in Table 3-1 and the instructions contained in SOP CTO0020-010 (Appendix A.1). Perform at least one calibration/standardization check daily. Record the total PCB concentration in the field log. QC checks for field analyses shall be conducted at the frequencies indicated in the test kit instructions and shall meet the specifications of the test kit. The information to be recorded includes but is not limited to the following:

- Analyst's name
- Date/time of analysis
- Test kit identifier, including serial number, if provided
- Reference to corresponding calibration/standardization check
- Sample number
- Measured concentration, including the measurement units

Report the total PCB concentration to the EMAC and the Navy so a decision can be made concerning the need for continued excavation.

As long as confirmation samples are being produced with field-measured total PCB concentrations greater than 1 mg/kg in surface samples or 25 mg/kg in subsurface samples, 1 of 10 composite samples will be sent for laboratory verification of PCB concentrations (as Aroclors). In this case, surface soil samples are samples collected within 2 feet of original grade as estimated through visual observation and simple field measurement with a tape measure or other suitable device. If any sample at the limit of excavation has a total PCB lead concentration less 1 mg/kg (surface soils) or 25 mg/kg (subsurface soils), the sample will be sent to the laboratory for verification of PCBs. This will ensure that a minimum of 10 percent or more of field analyses and all final results supporting the conclusion to terminate excavation are verified with laboratory results. For any sample analyzed in the field and in the fixed-base laboratory, both the field and the laboratory analyzed portions will be labeled with the same number in accordance with SOP COP0020-02 (Appendix A.1).

Sediment Waste Characterization

The goal of waste characterization sampling is to characterize sediments that will be disposed at an off-site disposal facility to determine whether the waste sediments meet the facility's waste acceptance criteria. Details of sediment waste characterization are being resolved by Navy and TtNUS and **will** be incorporated after they have been finalized.

Wastewater Characterization

Wastewater characterization requirements are being finalized by the Navy and TtNUS but a draft approach is presented here.

There are no waste water characterization requirements because PCBs are assumed to be entrained in sediments. Sediments from water collection tanks must be drained from the tanks and combined with other waste sediments for disposal.

3.6 QUALITY CONTROL REQUIREMENTS

Refer to TtNUS (2004) for generic QC requirements. Project-specific requirements are provided in this section.

In addition to instrument- or test kit-specific calibration requirements (see Section 3.4), the following field QA/QC samples will be collected at the indicated frequencies to support confirmation sampling and analyses.

- In general, duplicate confirmation samples will be collected at a rate of one for every 20 regular samples and analyzed for the same analytes as the corresponding regular samples. For SWMU 8, every fifth pond bank sample will be analyzed in duplicate for VOCs.

- When fixed-base laboratory analyses are used, enough additional sample volume will be collected to support MS/MSD sample analyses (organic chemical analyses only). The rate of MS/MSD analysis will be one for every 20 regular samples.

- Trip blanks must be included in each cooler containing samples submitted for laboratory confirmation of VOC analyses.

- A temperature blank will be added to each cooler that contain samples which must be kept cooled as a preservation requirement.

Definitions of each of these QC sample types are presented in TtNUS (2004). Collection of QC samples will not be required for waste characterization sampling and analyses unless required by the off-site disposal facility.

Equipment rinsate blanks (rinsate blanks) will not be collected because cross-contamination of confirmation samples or waste disposal samples is not a concern for this project. Source water blanks will not be collected because cross-contamination from source water is not a concern.

3.7 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING

Generic data reduction, validation, assessment and reporting requirements are provided in Section 8.0 of TtNUS (2004). Project-specific instructions are provided below for this project.

3.7.1 Confirmation Analysis Data

Data from field analyses and other field measurements must be recorded at the time of generation in the field log book or, when provided in the governing SOP, on the SOP-specific data forms. Confirmation sampling results will be entered into the TtNUS electronic database soon enough after the data collection to prevent loss of data and to support the generation of reports to the Navy. The data entry process will make use of a dual entry system whereby two independent data entry clerks enter the data and compare the results. Discrepancies, if detected, will be investigated to determine the correct values, and the correct values will be entered into the database. Non-discrepant results will be considered accurate unless subsequent data review or use indicates otherwise.

The data will be transferred to the Navy in accordance with requirements to be found at the following web site:

https://www.sdirport.com/customer/ttnus/irgateway.nsf/MAINLOADER?OpenFrameSet

The password required to access this site may be obtained through the Navy RPM. The information at this link provides detailed instructions for reporting environmental data. The intent is to standardize reporting formats to improve how environmental data are acquired, managed, and transmitted. The

following items are available at this link and the Electronic Data Exchange (EDE) Examples button located at the link:

- SOP
- Electronic Data Deliverable (EDD) spreadsheets and databases
- Electronic data processor
- Example electronic data submittal

Fixed-base laboratory verification data may be compared to field analytical data to determine whether there is a reasonable correlation between the field and fixed-base laboratory data. This will be done by plotting the laboratory data versus field data in a correlation plot. A correlation coefficient of 0.65 or greater and an apparent linear relationship (determined through visual inspection of the plotted data) will be considered a reasonably strong correlation. If a correlation of this degree is obtained, the field data may be used to estimate the equivalent laboratory concentration. However, fixed-base laboratory data shall be used as the definitive data to judge whether IMs have been completed satisfactorily.

3.7.2 Waste Characterization Data

Waste characterization analyses will be provided to the waste disposal facility and NAVFAC SE in the form of hard copies of laboratory data packages as well as data summaries. The data summaries shall identify each sample analyzed, the laboratory conducting the analysis, the results of analysis, and any qualifying notes indicating limitations on data use. At the Navy's discretion, data may also be provided in electronic format in accordance with the specifications identified in Section 3.7.1.

3.8 MANAGEMENT OF INVESTIGATION-DERIVED WASTE

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

- Personal protective equipment (PPE)
- Equipment decontamination fluids
- Waste soil from sampling activities

IDW will be handled as described in SOP CTO0020-09 (Appendix A.1).

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

Separate HASPs will be prepared by TtNUS and each EMAC. These HASPs describe specific health and safety requirements, concerns, and information related to the site activities. These documents will be read and understood by each person working at the site. Each worker or visitor to the site must sign an acknowledgment that he or she has read and understands the HASP.

3.9.2 Health and Safety Training

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

3.9.3 Medical Approval and Personal Protective Clothing and Equipment

TtNUS 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 the SSO for each contractor feels it is appropriate. The SSO will discuss safety issues related to activities being performed and will make site workers aware of any new conditions that could potentially affect health or safety.

3.10 PERFORMANCE AND SYSTEM AUDITS

Section 9.1 of the SWMU 8 and 15 QAPP (TtNUS, 2004) presents generic requirements for field operations performance and system audits. No special requirements are anticipated for this project.

3.11 INSPECTION AND MAINTENANCE

Sections 10.1 and 10.3 of TtNUS (2004) present generic inspection and maintenance requirements to ensure that field consumables and equipment are in proper working order and available when needed. In addition, project-specific requirements are provided below.

Chemical Test Kits - PCBs

The receipt date and the expiration date of receipts (i.e., purchase date plus 1 year) shall be written on the test kit (receipt date) and the reagents (expiration date) on each reagent bottle and the kit if the expiration date is not already indicated. Follow manufacturer instructions for storage and handling of the kits. Do not use kits on kit components past their expiration dates.

XRF Instrument

Ensure that the lead calibration coupon (calibration check standard) is available for routine calibration checks that must be conducted periodically on each day of use. The operability of the instrument should be checked the day before the beginning of use.

Color-Tec Apparatus

Ensure that the color tubes have been stored in accordance with manufacturer directions and that enough tubes are available for each day of use. There should be more than one tube for each sample to be analyzed for each anticipated concentration level expected to be encountered at the SWMU. Inspect the vacuum apparatus prior to use to ensure that it there are no visible leaks in the vacuum line.

3.12 FIELD CORRECTIVE ACTIONS

Field nonconformances or conditions adverse to quality must be identified and corrected as quickly as possible so that work integrity or product quality is not compromised. The need for corrective action may arise based on deviations from project plans and procedures, adverse field conditions, or other unforeseen circumstances. Corrective action needs may become apparent during the performance of daily work tasks or as a consequence of internal or external field audits.

Corrective action may include resampling, re-excavation or other rework and may involve amending previously approved field procedures. Minor modifications to field activities, such as the collection of additional samples, will be initiated at the discretion of the FOL, subject to on-site approval by NSWC

Crane personnel. Major modifications or situations that affect compliance with or achievement of Data Quality Objectives (DQOs) must be approved and documented via a Field Task Modification Request (FTMR). An example FTMR is provided as Figure 3-12. Approval of the corrective action will be obtained by the United States Navy (in conjunction with IDEM). The TtNUS or EMAC FOL is responsible for initiating FTMRs. An FTMR will be prepared for all deviations from the project plan documents, as applicable. Copies of all FTMRs will be maintained with the on-site project planning documents and will be placed in the final evidence file.

3.13 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The TtNUS FOL will provide the TOM with daily verbal field progress reports during the course of the IMs. The EMAC FOL will provide daily verbal reports to the EMAC PM during the course of IMs. These reports will explain accomplishments, deviations from the QAPP, and upcoming activities, and will provide a QA summary. The TtNUS TOM and EMAC PM will provide monthly progress reports to the Navy that address the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.

TABLE 3-1

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR FIELD ANALYSIS AND LABORATORY CONFIRMATION ANALYSIS OF SOILS AND SEDIMENTS
SWMUS 7, 8, 13, AND 17 INTERIM MEASURES
NSWC CRANE, CRANE, INDIANA

Parameter	Applicability	Analyte/Methodology/ SOP	Sample Container	Container Volume	Preservation	Maximum Holding Time[1]
Field Analyses						
Total Lead	SWMU 7 only	Field XRF (SOP CTO020-07)	Thin-walled plastic sandwich bag	8-ounce	NA	Analysis within 180 days
PCBs	SMWUs 13 and 17	RaPID Assay Test Kit (0.5 to 10 ppm) with SDI Sample Extraction Kit	Wide-mouth jar, Teflon-lined plastic cap	8-ounce	NA	Analysis within 14 days
Volatile Organic Compounds	SMWU 8 only	Color-Tec Apparatus with GasTech color tubes (SOP CTO020-08)	Wide-mouth jar, Teflon-lined plastic cap	Three 5-gram	Cool to 4 °C	3 Hours to analysis
Laboratory Analyses [2]						
Total Lead	SWMU 7 only	SW-846 3050B/6020 LTL-7015/ LTL-7202	Wide-mouth jar, Teflon-lined plastic cap	8-ounce	NA	Analysis within 180 days
PCBs	SMWUs 13 and 17	SW-846 8082 LTL-3302 / LTL-8084	Wide-mouth jar, Teflon-lined plastic cap	8-ounce	Cool to 4 °C	Extraction within 14 days; analysis within 40 days of extraction.
Volatile Organic Compounds	SMWU 8 only	SW-846 8260B (5-gram purge) LTL-8265	3 Encore™ samplers [3]	Three 5-gram	Cool to 4 °C	48 hours to preservation; 14 days to analysis.

1 All holding times are from date of collection. Laboratory analysis will be 1-day turnaround.
2 For parameter lists for PCBs and VOCs, see the SWMU 8 and 15 QAPP, Table 1-5. That table also has target detection and quantitation (reporting) limits.
3 One Encore™ extruded into 5 mL water and frozen. Second Encore™ frozen as received. Third Encore™ preserved with methanol for medium-level analysis.
°C - Degrees Centigrade.
LTL - Laucks Testing Laboratories, Inc.

TABLE 3-1

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR FIELD ANALYSIS AND LABORATORY CONFIRMATION ANALYSIS OF SOILS AND SEDIMENTS SWMUS 7, 8, 13, AND 17 INTERIM MEASURES NSWC CRANE, CRANE, INDIANA

PCBs - Polychlorinated biphenyls.
ppm - Parts per million.
SOP - Standard operating procedure
SW-846 - U.S. EPA, 1986. Test methods for Evaluating Solid Wastes, Physical/Chemical Methods. SW-846, Third Edition and subsequent updates.
XRF - X-ray flourescence spectroscopy.
VOCs - Volatile organic compounds.

TABLE 3-2

SUMMARY OF SAMPLE ANALYSES, CONTAINER TYPES AND VOLUMES, PRESERVATION REQUIREMENTS, AND HOLDING TIMES FOR FIELD ANALYSIS AND LABORATORY ANALYSIS OF AQUEOUS SAMPLES SWMUs 7, 8, 13, AND 17 INTERIM MEASURES NSWC CRANE, CRANE, INDIANA

Parameter	Applicability	Analyte/Methodology	Sample Container	Container Volume	Preservation	Maximum Holding Time[2]
Field Analysis						
Volatile Organic Compounds	SMWU 8 only™	Color-Tec Apparatus with GasTech color tubes (CTO0020-08)	Glass, black phenolic plastic screw cap, Teflon-lined septum	Three 40 mL	Cool to 4 °C, zero headspace; **DO NOT USE HYDROCHLORIC ACID**	3 Hours to analysis
Laboratory Analysis [1]						
Volatile Organic Compounds	SMWU 8 only	SW-846 8260B (25 mL purge) LTL-8265 (low level)	Glass, black phenolic plastic screw cap, Teflon-lined septum	Three 40 mL	Cool to 4 °C, dark, zero headspace, HCl to pH <2	14 days to analysis.

1 For parameter lists for PCBs and VOCs, see the SWMU 8 and 15 QAPP, Table 1-5. That table also has target detection and quantitation limits.

2 All laboratory holding times are from date of collection; field analysis holding times are from time of collection. Laboratory anlaysis will be 1-day turnaround.

°C - Degrees Celsius
HCl - Hydrochloric acid.
LTL - Laucks Testing Laboratories, Inc.
mL - Milliliter.
PCBs - Polychlorinated biphenyls.
SW-846 - U.S. EPA, 1986. Test methods for Evaluating Solid Wastes, Physical/Chemical Methods. SW-846, Third Edition and subsequent updates.

TABLE 3-3

VERIFICATION SAMPLE LOCATIONS FOR SWMU 7 EXCAVATION FLOOR AND SIDE WALL SAMPLES
SWMUs 7, 8, 13, AND 17 INTERIM MEASURES
NSWC CRANE, CRANE, INDIANA

Set #1 - Excavation Floor and Interim Measures Support Facility Composite Samples for SWMUs 7, 13, and 17

Sample Rotation	Grid Location		Sample Rotation	Grid Location		Sample Rotation	Grid Location		Sample Rotation	Grid Location	
	X %	Y %		X %	Y %		X %	Y %		X %	Y %
Sample 1	93	80	Sample 6	19	61	Sample 11	5	62	Sample 16	28	53
	80	27		93	93		21	33		76	43
	61	69		76	13		57	17		6	56
	7	66		52	20		48	90		53	91
Sample 2	89	69	Sample 7	99	4	Sample 12	13	2	Sample 17	43	85
	67	53		35	5		78	62		16	91
	38	34		51	3		88	78		73	92
	63	67		92	22		53	96		37	5
Sample 3	30	70	Sample 8	39	65	Sample 13	92	54	Sample 18	34	34
	74	70		12	10		90	97		49	2
	23	30		83	56		41	57		45	18
	36	40		73	73		26	88		5	18
Sample 4	66	98	Sample 9	35	87	Sample 14	34	5	Sample 19	29	42
	70	92		34	64		35	5		46	88
	25	42		42	25		82	68		86	64
	69	40		51	29		54	95		35	21
Sample 5	76	44	Sample 10	59	1	Sample 15	82	79	Sample 20	28	42
	96	49		52	63		2	40		17	8
	0	14		18	20		50	91		19	27
	69	73		50	58		11	68		56	1

Notes: 1) For SWMU 17, where excavtion floor is bedrock, no verification sample will be collected.
2) One verification sample will be collected for every 100 feet of access road/trail length.

Set #2 - Excavation Wall / Bank Composite Samples for SWMUs 7, 13, and 17

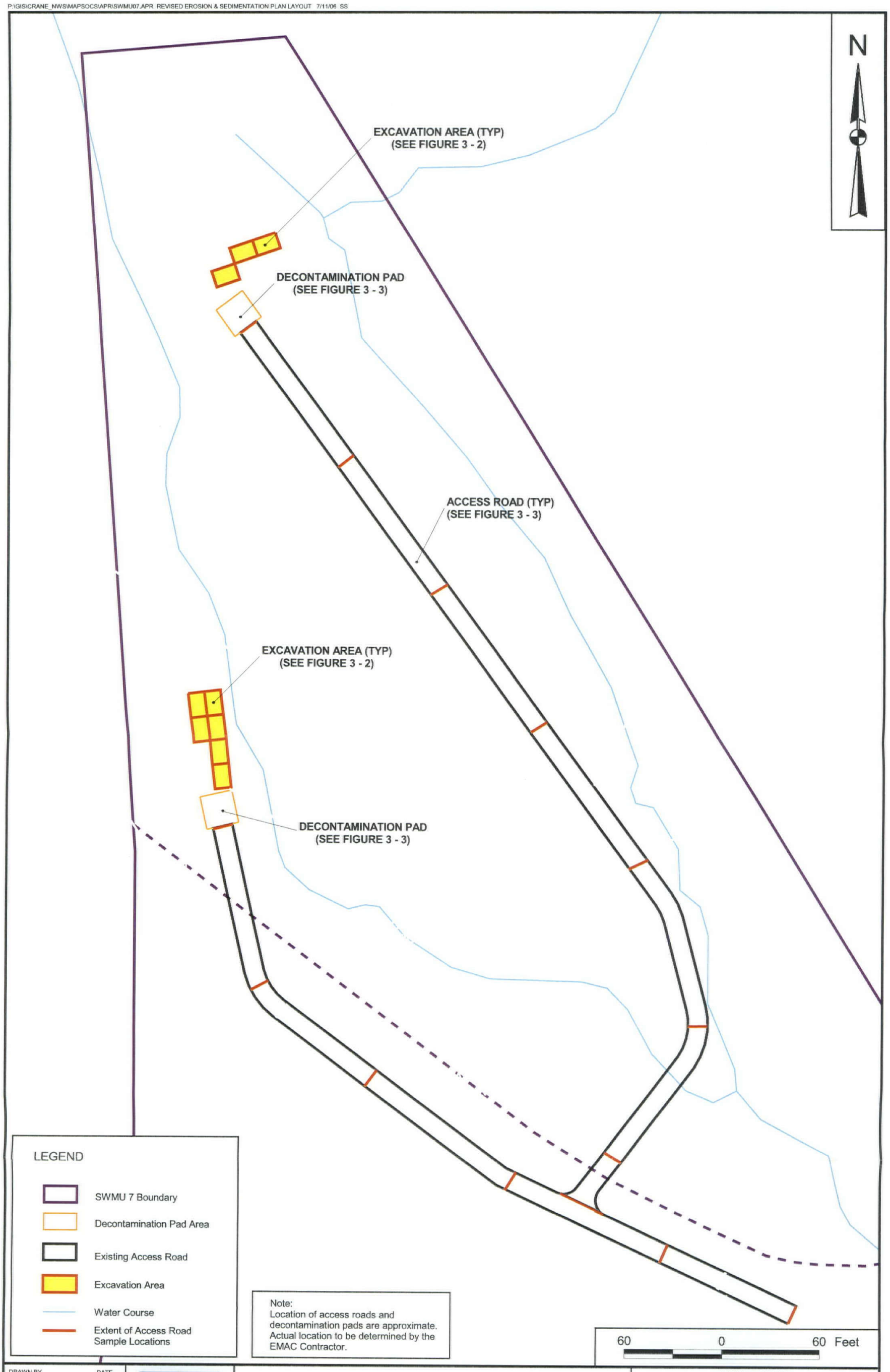
Sample Rotation	Grid Location		Sample Rotation	Grid Location		Sample Rotation	Grid Location		Sample Rotation	Grid Location	
	X %	Y %		X %	Y %		X %	Y %		X %	Y %
Sample 1	17	8	Sample 6	23	30	Sample 11	46	85	Sample 16	90	25
	5	15		90	19		9	32		56	76
Sample 2	67	83	Sample 7	83	51	Sample 12	26	52	Sample 17	85	62
	30	58		67	12		66	52		16	82
Sample 3	55	12	Sample 8	87	23	Sample 13	63	81	Sample 18	48	96
	89	77		66	29		66	74		23	74
Sample 4	51	8	Sample 9	82	95	Sample 14	51	92	Sample 19	48	92
	66	57		64	36		53	9		89	13
Sample 5	17	64	Sample 10	1	2	Sample 15	93	79	Sample 20	17	91
	45	56		30	98		81	41		63	71

Set #3 - Excavation Wall and Interim Measures Support Facility Grab Samples for SWMU 8

Sample Rotation	Grid Location		Sample Rotation	Grid Location		Sample Rotation	Grid Location		Sample Rotation	Grid Location	
	X %	Y %		X %	Y %		X %	Y %		X %	Y %
Sample 1	19	86	Sample 6	10	97	Sample 11	48	76	Sample 16	74	41
Sample 2	72	73	Sample 7	91	64	Sample 12	83	28	Sample 17	31	37
Sample 3	16	61	Sample 8	72	21	Sample 13	45	10	Sample 18	15	0
Sample 4	77	61	Sample 9	95	19	Sample 14	48	97	Sample 19	64	95
Sample 5	32	8	Sample 10	16	45	Sample 15	6	19	Sample 20	87	63

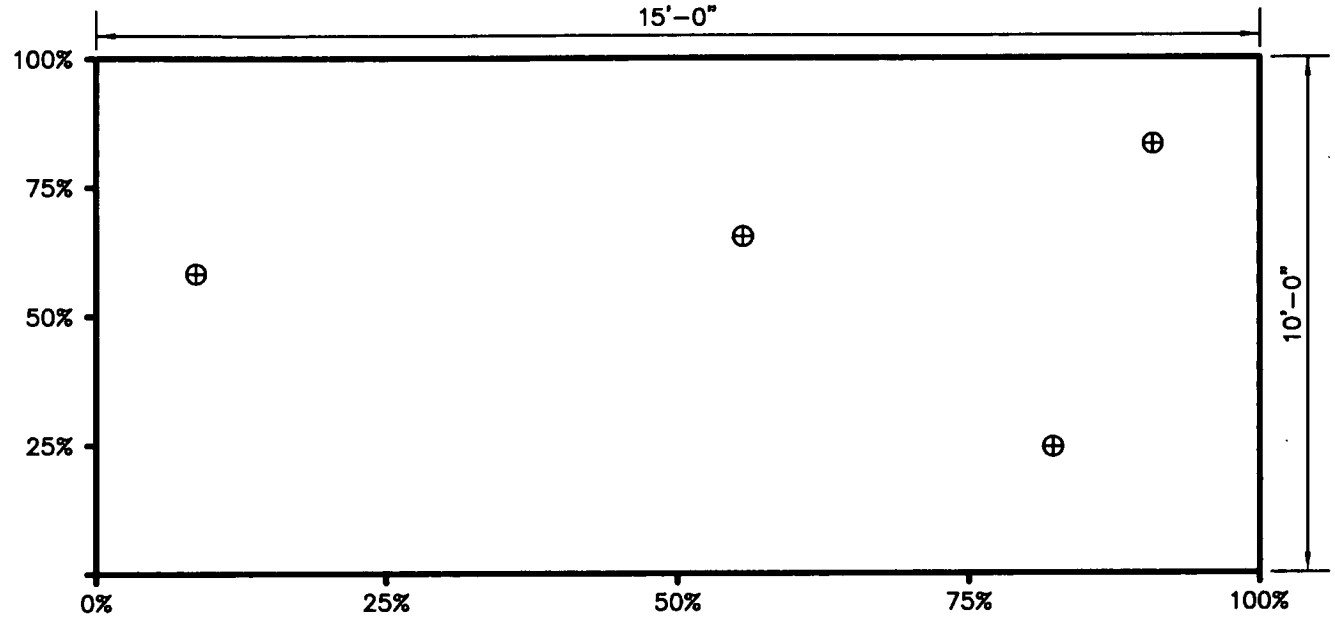
Notes: 1) For SWMU 8, because excavtion floor will be bedrock, no verification sample will be collected on the excavation floor.
2) One verification sample will be collected at a rate of one verification samples per 500 square feet of support facility eatablised, and at a rate of one verification sample per 25 feet of access road.

P:\GIS\CRANE_NWS\MAPSOCS\APR\SWMU07.APR REVISED EROSION & SEDIMENTATION PLAN LAYOUT 7/11/06 SS

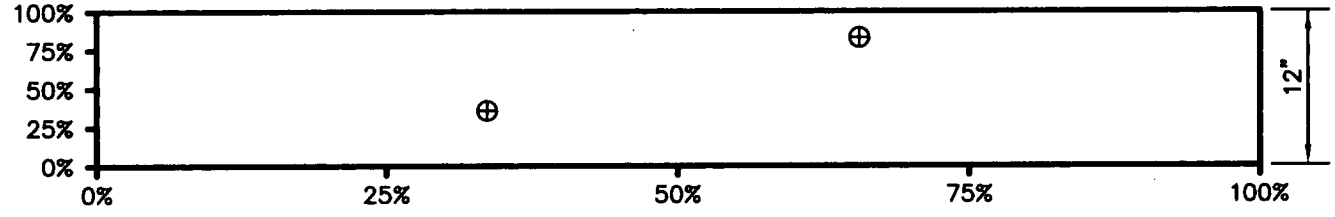


DRAWN BY S. STROZ	DATE 6/15/06	SWMU 7 - VERIFICATION SAMPLING - EXCAVATION AREAS AND SUPPORT FACILITIES INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NUMBER CTO 0020
CHECKED BY T. SMITH	DATE 7/11/06		APPROVED BY — DATE —
COST/SCHEDULE-AREA			APPROVED BY — DATE —
SCALE AS NOTED	NAVFAC		DRAWING NO. FIGURE 3 - 1 REV 0

ACAD: 0352CP01.dwg 07/12/06 DT PIT



TYPICAL SWMU 7 EXCAVATION FLOOR
LOCATIONS REPRESENT SAMPLE 1, SET 1, ON TABLE 3-3



TYPICAL SWMU 7 EXCAVATION SIDE WALL
LOCATIONS REPRESENT SAMPLE 2, SET 2, ON TABLE 3-3

LEGEND:

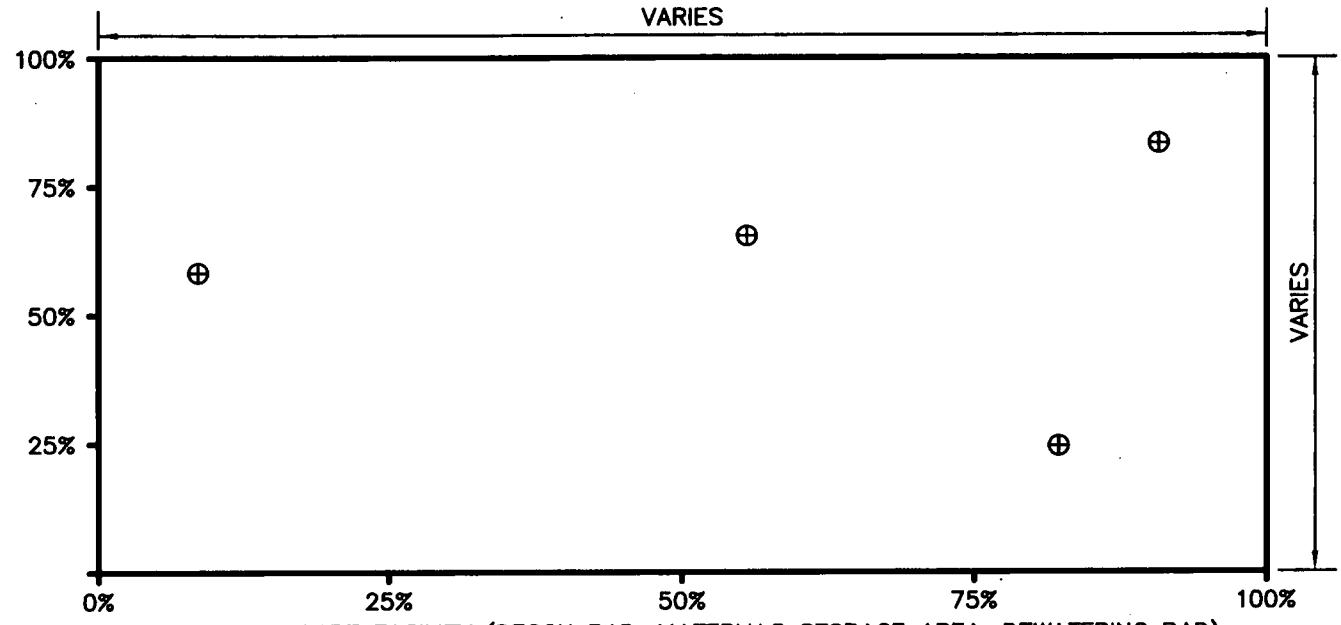
⊕ ALIQUOT SAMPLE LOCATION

0% PERCENT DISTANCE ALONG EXPOSED SURFACE

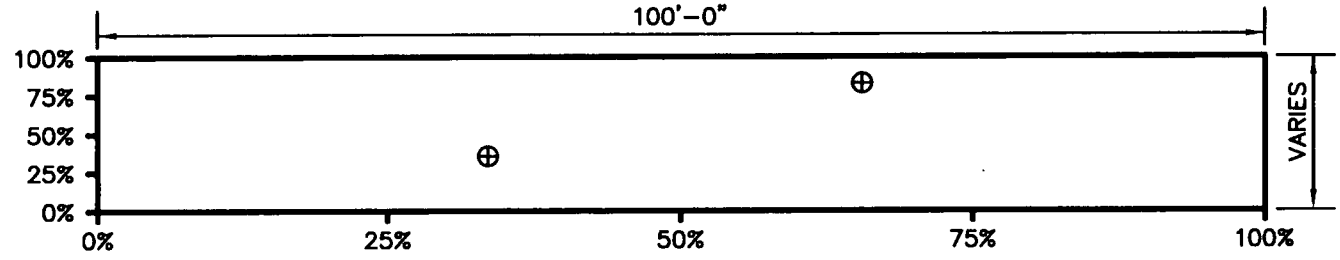
DRAWN BY MF	DATE 6/6/06	SWMU 7 EXCAVATION VERIFICATION SAMPLE LOCATIONS EXAMPLE INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-2 / REV. 0

FORM CADD NO. SDIV-AH.DWG - REV 1 - 9/10/98

ACAD: 0352CP02.dwg 07/12/06 DT PIT



TYPICAL SUPPORT FACILITY (DECON PAD, MATERIALS STORAGE AREA, DEWATERING PAD)
LOCATIONS REPRESENT SAMPLE 1, SET 1, ON TABLE 3-3



TYPICAL SITE ACCESS ROAD/TRAIL
LOCATIONS REPRESENT SAMPLE 2, SET 2, ON TABLE 3-3

LEGEND:

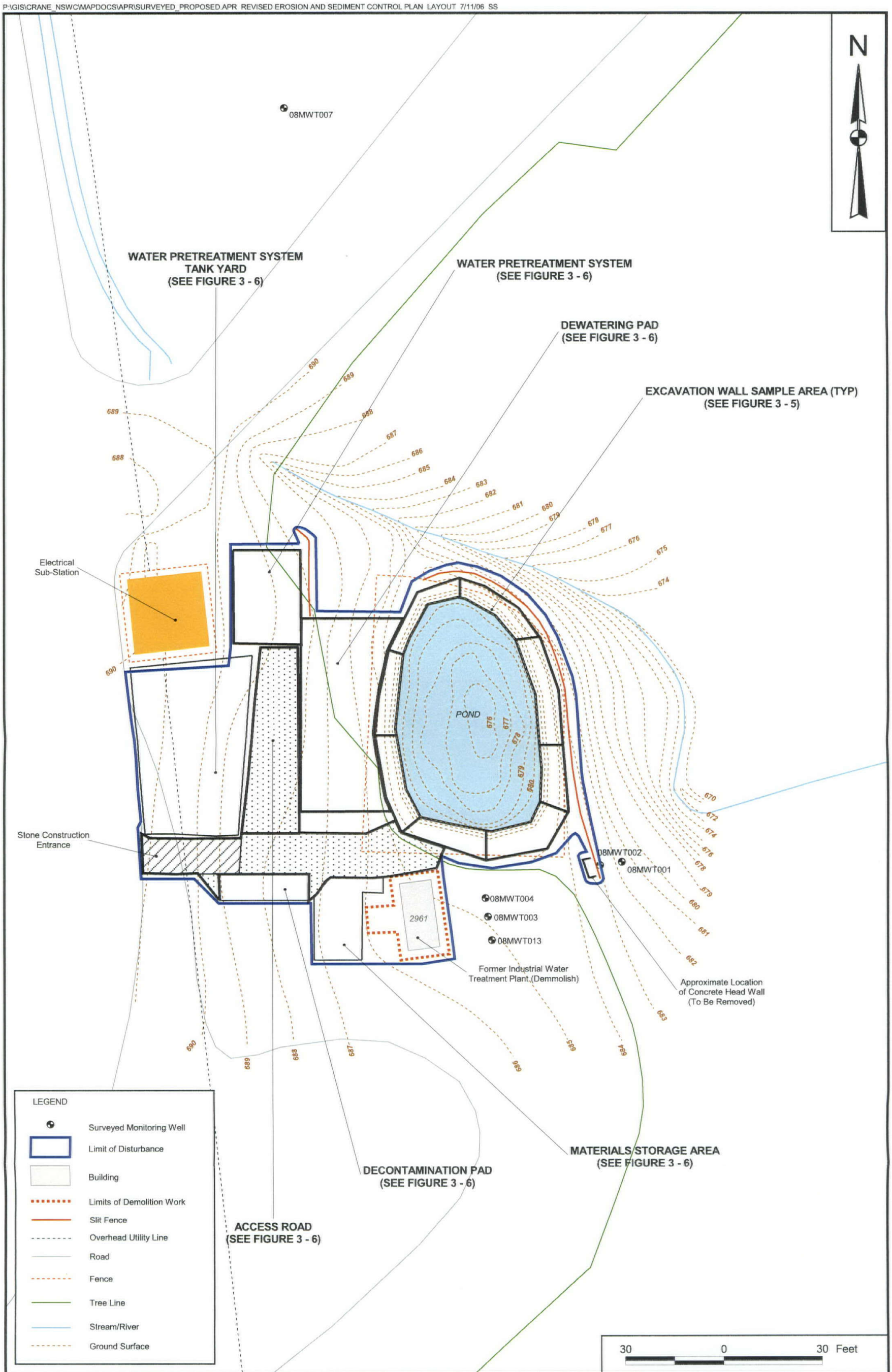
⊕ ALIQUOT SAMPLE LOCATION

0% PERCENT DISTANCE ALONG EXPOSED SURFACE

DRAWN BY MF	DATE 6/6/06	SWMU 7 INTERIM MEASURES SUPPORT FACILITY COMPOSITE SAMPLING EXAMPLE INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-3 / REV. 0

FORM CADD NO. SDIV-AH.DWG - REV 1 - 9/10/98

P:\GIS\CRANE_NSWC\MAPDOCS\APR\SURVEYED_PROPOSED.APR REVISED EROSION AND SEDIMENT CONTROL PLAN LAYOUT 7/11/06 SS



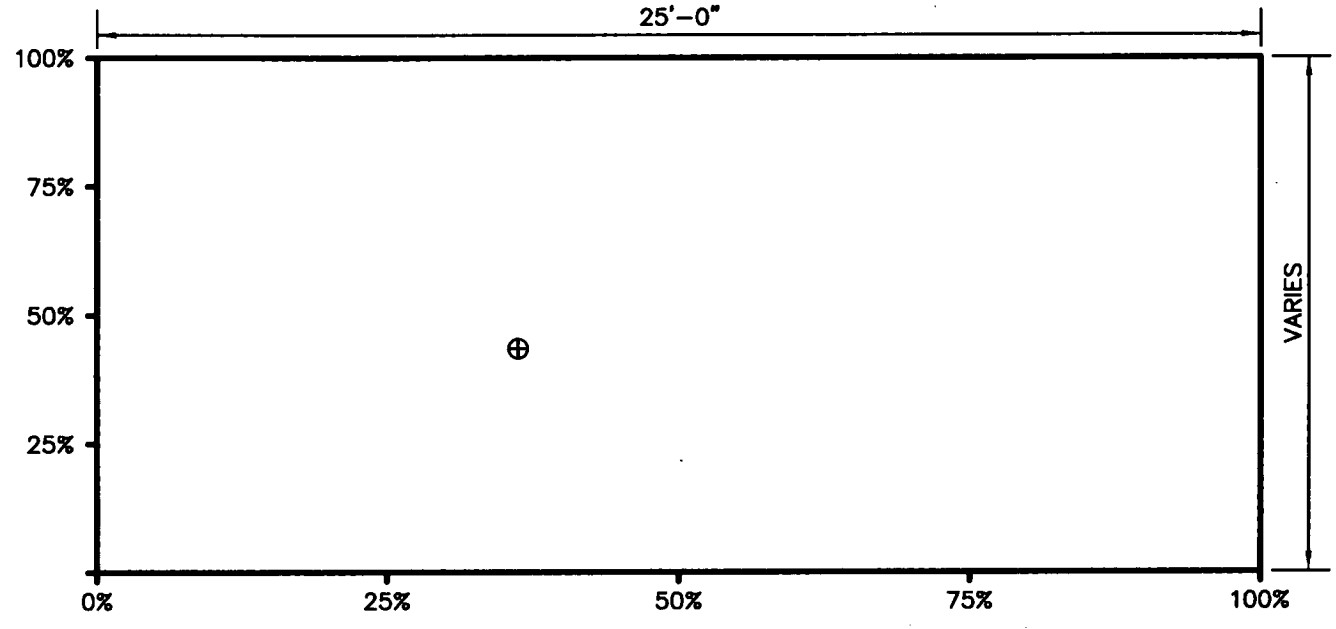
DRAWN BY	DATE
S. STROZ	6/14/06
CHECKED BY	DATE
T. JOHNSTON	7/11/06
COST/SCHEDULE-AREA	
SCALE	
AS NOTED	

NAVFAC

SWMU 8 VERIFICATION SAMPLING - EXCAVATION
AREAS AND SUPPORT FACILITIES
INTERIM MEASURES QAPP ADDENDUM NO. 1
NSWC CRANE
CRANE, INDIANA

CONTRACT NUMBER	
CTO 0020	
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
DRAWING NO.	REV
FIGURE 3 - 4	0

ACAD:0352CP03.dwg 06/12/06 MF PIT



SWMU 8 POND EXCAVATION WALL
(TYPICAL)

LEGEND:

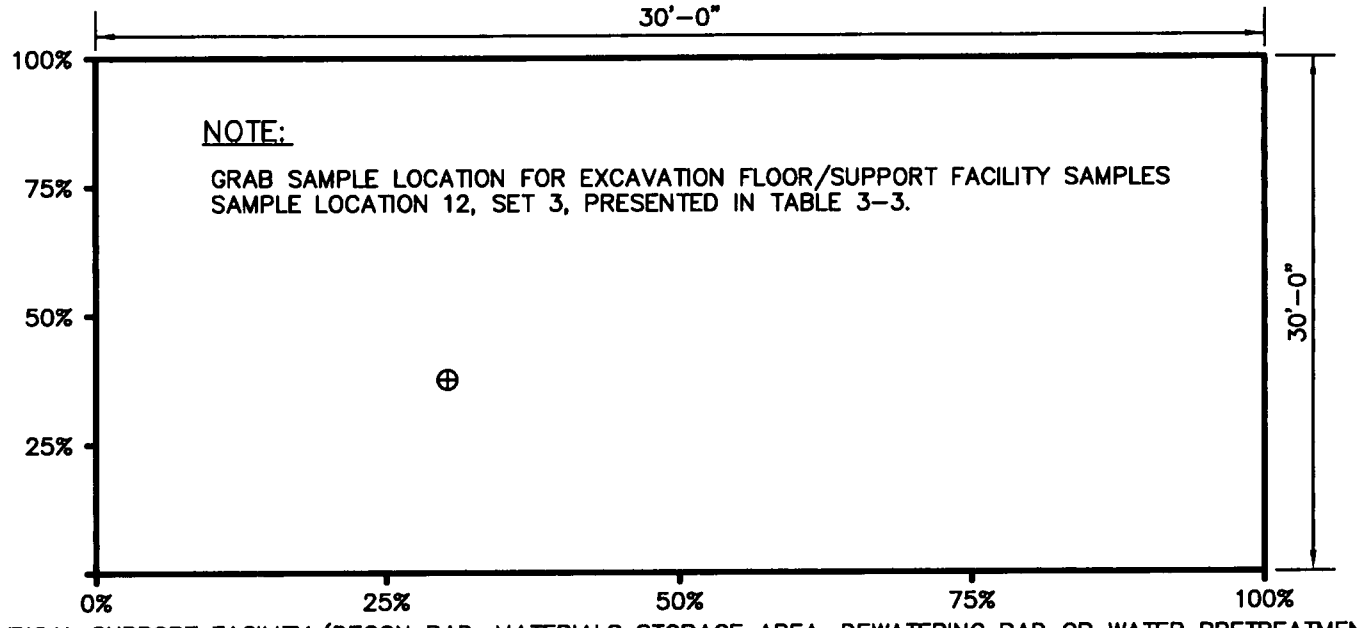
⊕ GRAB SAMPLE LOCATION. LOCATIONS PRESENTED IDENTIFY THE SAMPLE LOCATIONS FOR EXCAVATION WALL SAMPLE LOCATION 17, SET 3, PRESENTED IN TABLE 3-3.

0% PERCENT DISTANCE ALONG EXPOSED SURFACE.

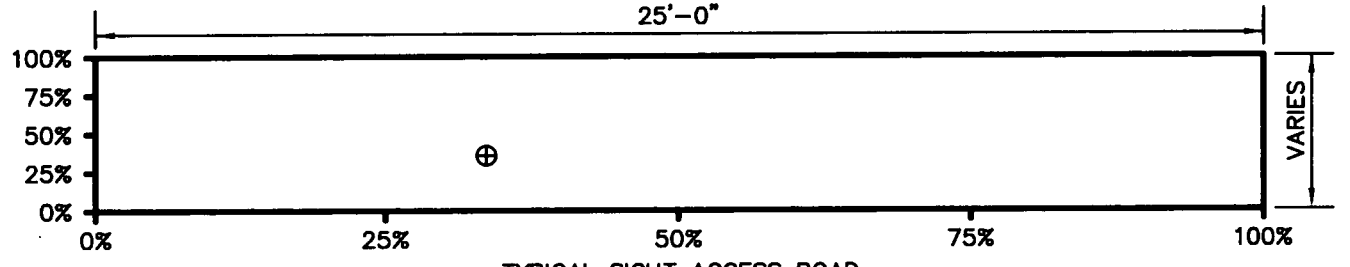
DRAWN BY MF	DATE 6/6/06	SWMU 8 INTERIM MEASURES EXCAVATION SIDEWALL VERIFICATION SAMPLE LOCATIONS INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-5 / REV. 0

FORM CADD NO. SDIV-AH.DWG - REV 1 - 9/10/98

ACAD: 0352CP04.dwg 07/12/06 DT PIT



TYPICAL SUPPORT FACILITY (DECON PAD, MATERIALS STORAGE AREA, DEWATERING PAD OR WATER PRETREATMENT
(ONE GRAB FOR EVERY 500 FT^2)



TYPICAL SIGHT ACCESS ROAD

LEGEND:

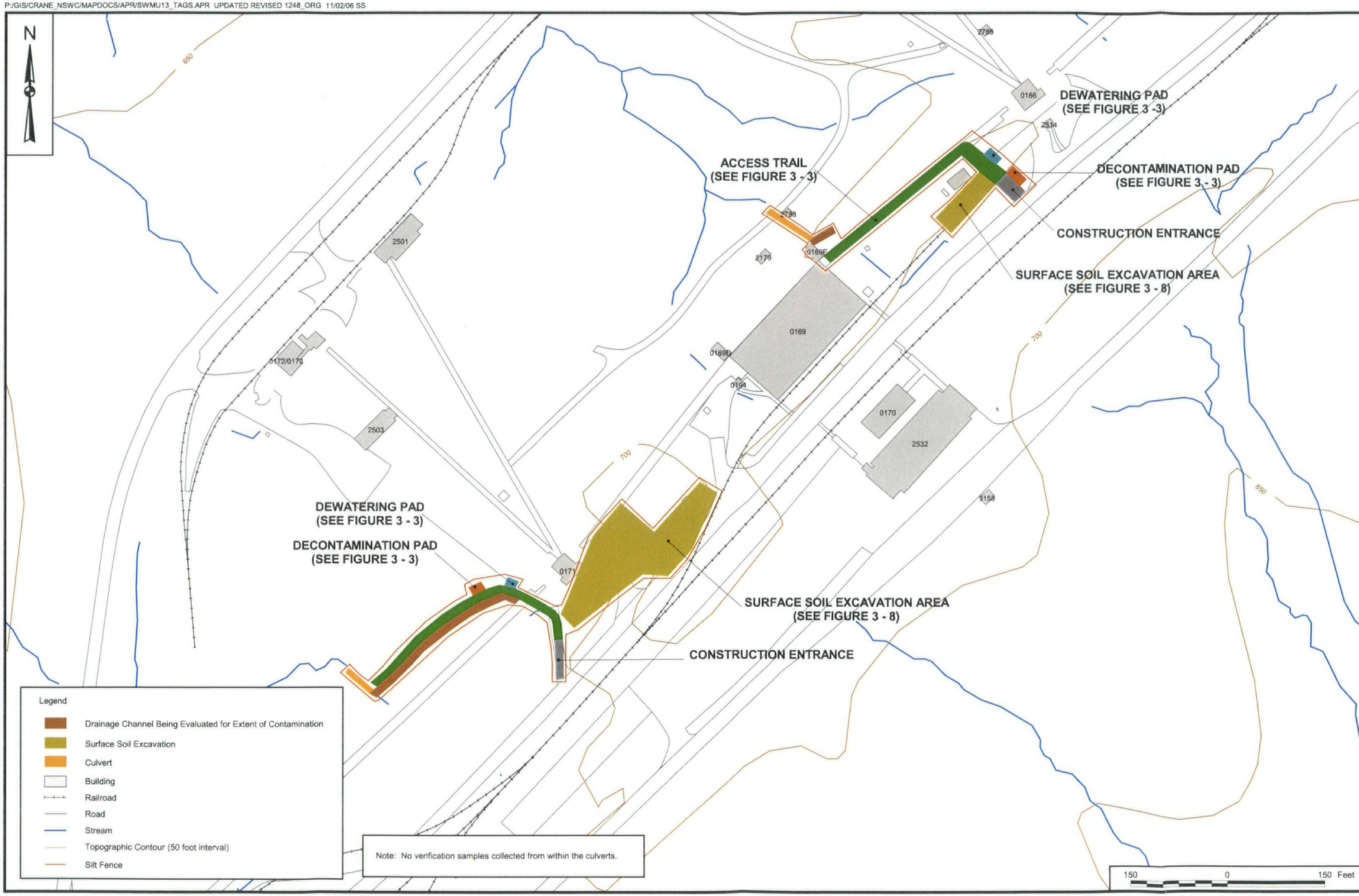
⊕ GRAB SAMPLE LOCATION (SEE NOTE)

0% PERCENT DISTANCE ALONG EXPOSED SURFACE

DRAWN BY MF	DATE 6/6/06	SWMU 8 INTERIM MEASURES SUPPORT FACILITY COMPOSITE SAMPLING EXAMPLE INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-6 / REV. 0

FORM CADD NO. SDIV-AH.DWG - REV 1 - 9/10/98

P:/GIS/CRANE_NSWC/MAPDOCS/APR/SWMU13_TAGS.APR UPDATED REVISED 1248_ORG 11/02/06 SS



CONTRACT NO. 0020

APPROVED BY ___ DATE ___

APPROVED BY ___ DATE ___

DRAWING NO. FIGURE 3 - 7

REV 0

VERIFICATION SAMPLING - EXCAVATION AREAS AND SUPPORT FACILITIES
SWMU 13 - MINE FILL B
INTERIM MEASURES QAPP ADDENDUM NO. 1
NSWC CRANE
CRANE, INDIANA

NAVFAC

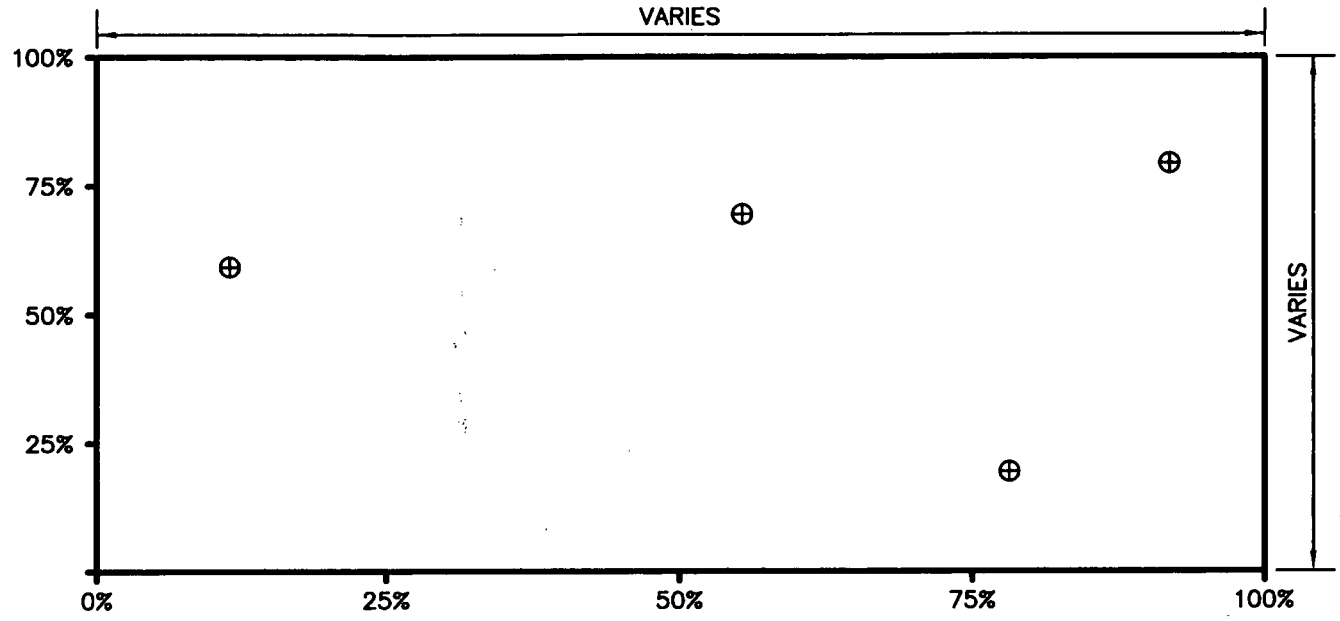
DRAWN BY S. STROZ DATE 7/12/06

CHECKED BY V. PLACHY DATE 11/02/06

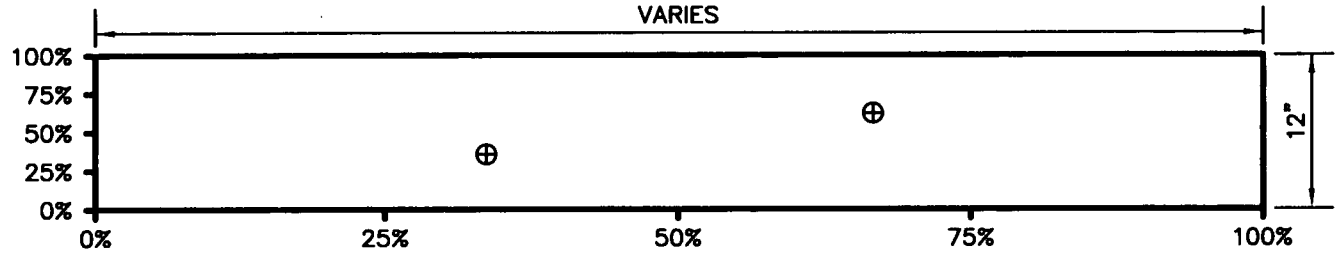
COST/SCHED-AREA

SCALE AS NOTED

ACAD: 0352CP05.dwg 06/12/06 MF PIT



TYPICAL SWMU 13 EXCAVATION FLOOR
LOCATION REPRESENTS SAMPLE 1, SET 1, ON TABLE 3-3



TYPICAL SWMU 13 EXCAVATION SIDEWALL
LOCATION REPRESENTS SAMPLE 2, SET 2, ON TABLE 3-3

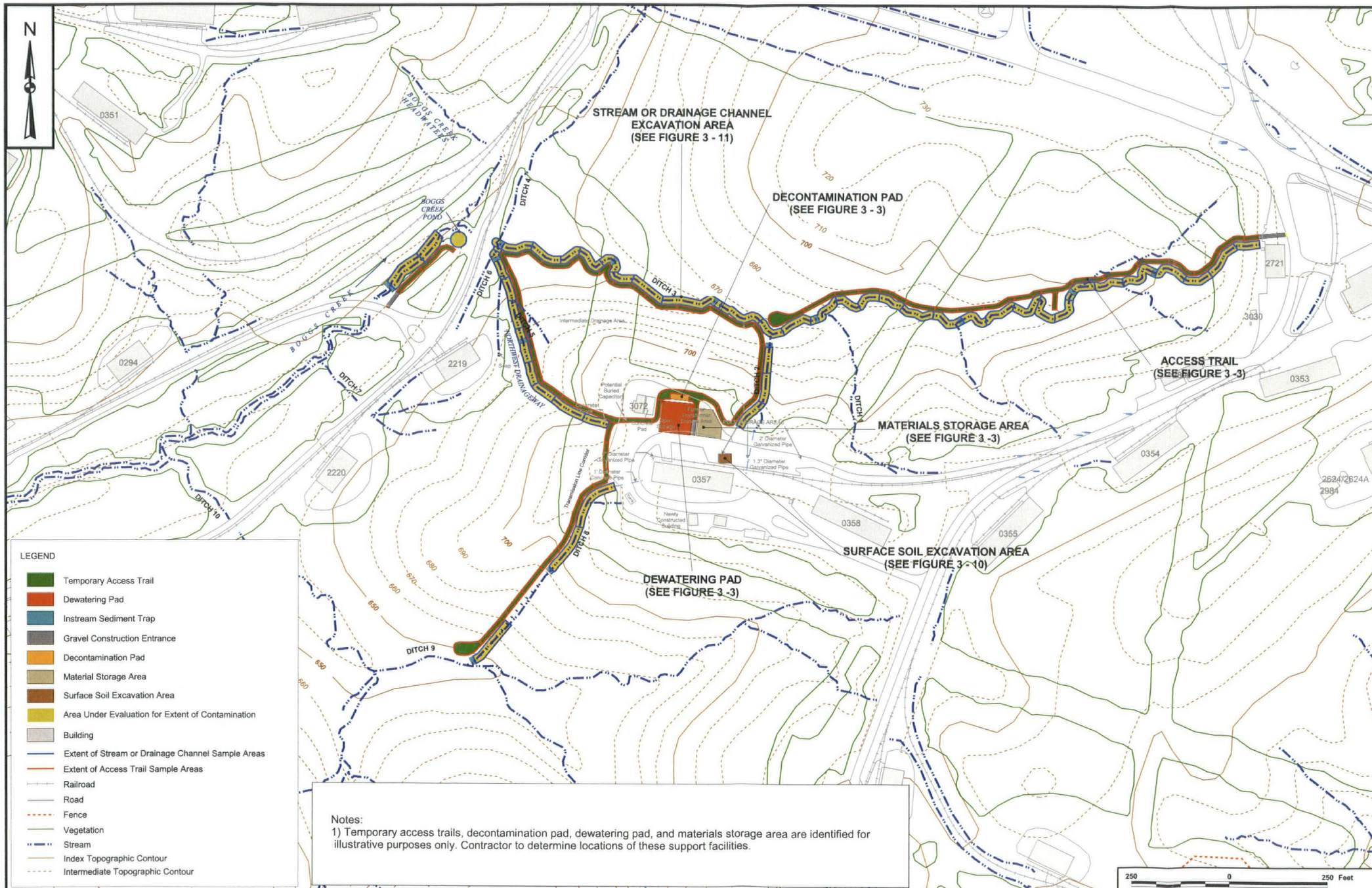
LEGEND:

⊕ ALIQUOT SAMPLE LOCATION

0% PERCENT DISTANCE ALONG EXPOSED SURFACE

DRAWN BY MF	DATE 6/6/06	SWMU 13 INTERIM MEASURES EXCAVATION VERIFICATION SAMPLE LOCATIONS EXAMPLE INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-8 / REV. 0

FORM CADD NO. SDIV-AH.DWG - REV 1 - 9/10/98



Notes:
1) Temporary access trails, decontamination pad, dewatering pad, and materials storage area are identified for illustrative purposes only. Contractor to determine locations of these support facilities.

CONTRACT NO. 0020

APPROVED BY ___ DATE ___

APPROVED BY ___ DATE ___

DRAWING NO. FIGURE 3 - 9

REV 0

SWMU 17 VERIFICATION SAMPLING - EXCAVATION AREAS AND SUPPORT FACILITIES
INTERIM MEASURES QAPP ADDENDUM NO. 1
NSWC, CRANE
CRANE, INDIANA

NAVFAC

DRAWN BY S. STROZ DATE 6/19/06

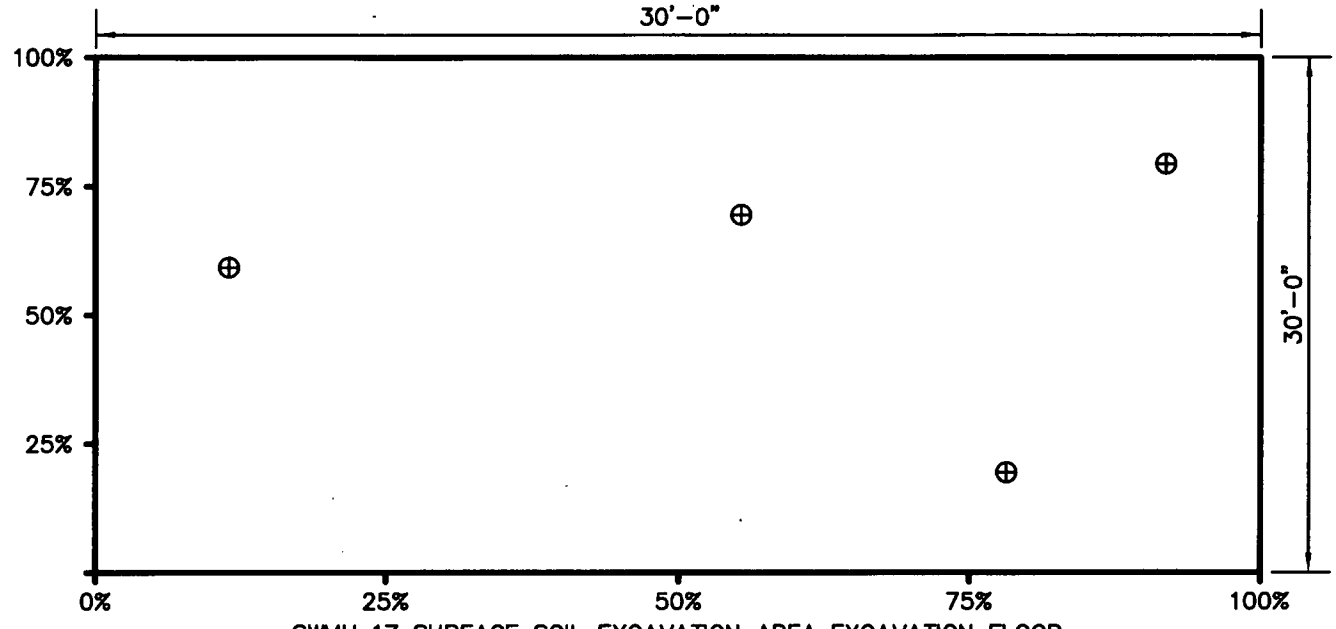
CHECKED BY V. PLACHY DATE 11/02/06

COST/SCHED-AREA

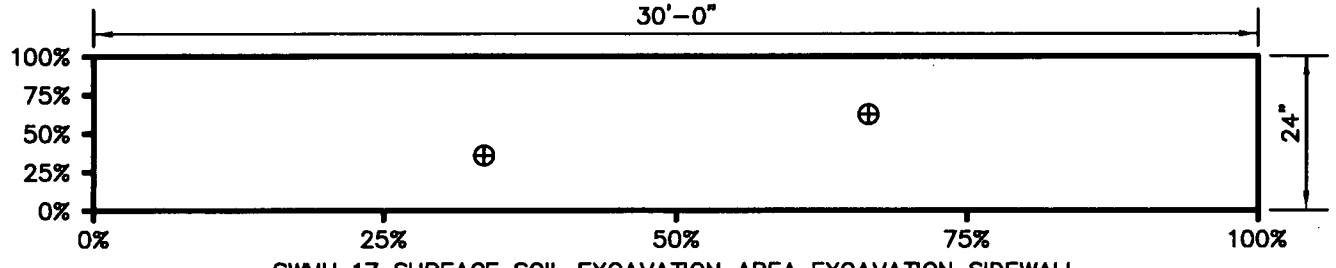
SCALE AS NOTED

P:\GIS\CRANE_NSWC\MAPDOCS\APR\SWMU17 APR REVISED SWMU 17 EROSION AND SEDIMENT CONTROL PLAN 11/02/06 SS

ACAD: 0352CP06.dwg 06/12/06 MF PIT



SWMU 17 SURFACE SOIL EXCAVATION AREA EXCAVATION FLOOR
LOCATION REPRESENTS SAMPLE 1, SET 1, ON TABLE 3-3



SWMU 17 SURFACE SOIL EXCAVATION AREA EXCAVATION SIDEWALL
LOCATION REPRESENTS SAMPLE 2, SET 2, ON TABLE 3-3

LEGEND:

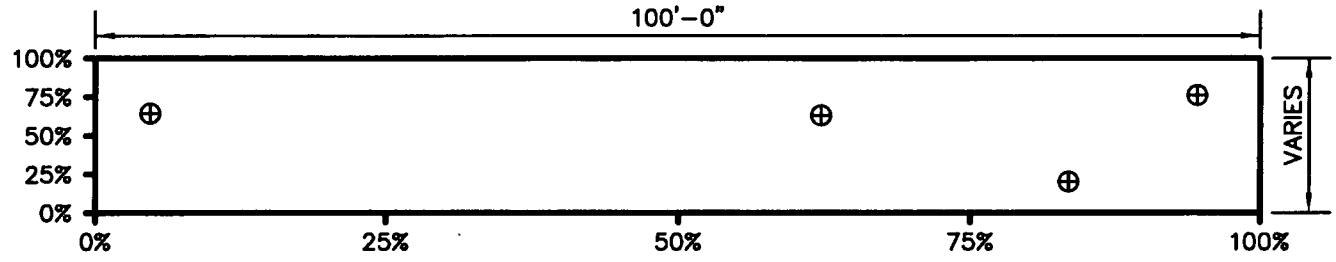
⊕ ALIQUOT SAMPLE LOCATION

0% PERCENT DISTANCE ALONG EXPOSED SURFACE

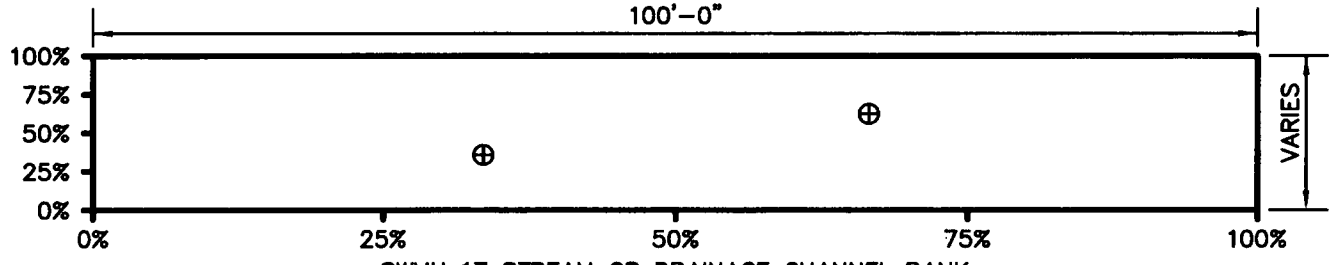
DRAWN BY MF	DATE 6/6/06	SWMU 17 SURFACE SOIL EXCAVATION AREA VERIFICATION SAMPLE LOCATIONS INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-10 / REV. 0

FORM CADD NO. SDIV-AH.DWG – REV 1 – 9/10/98

ACAD: 0352CP07.dwg 06/12/06 MF PIT



SWMU 17 STREAM OR DRAINAGE CHANNEL BED
LOCATION REPRESENTS SAMPLE 1, SET 1, ON TABLE 3-3



SWMU 17 STREAM OR DRAINAGE CHANNEL BANK
LOCATION REPRESENTS SAMPLE 2, SET 2, ON TABLE 3-3

LEGEND:

⊕ ALIQUOT SAMPLE LOCATION

0% PERCENT DISTANCE ALONG EXPOSED SURFACE

DRAWN BY MF	DATE 6/6/06	SWMU 17 STREAM AND DRAINAGE CHANNEL VERIFICATION SAMPLE LOCATIONS INTERIM MEASURES QAPP ADDENDUM NO. 1 NSWC CRANE CRANE, INDIANA	CONTRACT NO. 0352
CHECKED BY	DATE		OWNER NO.
REVISED BY	DATE		APPROVED BY / DATE
SCALE NONE		NAVFAC	DRAWING NO. FIGURE 3-11 / REV. 0

FORM CADD NO. SDIV-AH.DWG – REV 1 – 9/10/98

FIGURE 3-12

FIELD TASK MODIFICATION REQUEST FORM

Project/Installation Name _____ CTO & Project Number _____ Task Mod. Number _____

Modification To (e.g. Work Plan) _____ Site/Sample Location _____ Date _____

Activity Description: _____

Reason for Change: _____

Recommended Disposition: _____

Field Operations Leader (Signature) _____ Date _____

Approved Disposition:

Project/Task Order Manager (Signature) _____ Date _____

Distribution:

Program/Project File –
Project/Task Order Manager –
Field Operations Leader –

Other: _____

4.0 LABORATORY OPERATIONS AND DATA REDUCTION, REVIEW, AND MANAGEMENT

4.1 SAMPLE CUSTODY PROCEDURES

Samples will be labeled and shipped to the analytical laboratory in accordance with Section 3.0 of this document. Laboratory-specific custody procedures are governed by Section 4.0 of the SWMUs 8 and 15 QAPP (TtNUS, 2004).

4.2 LABORATORY INSTRUMENT CALIBRATION PROCEDURES AND FREQUENCIES

Refer to Section 5.2 of the SWMUs 8 and 15 QAPP (TtNUS, 2004) for laboratory calibration requirements. Only requirements for lead, PCB (as Aroclors) and VOC analyses will be applicable.

4.3 LABORATORY ANALYTICAL AND MEASUREMENT PROCEDURES

4.3.1 Confirmation Sample Analyses

The laboratory analyses of confirmation samples will be performed by Laucks Testing Laboratories. Laucks Testing Laboratories has successfully completed the laboratory evaluation process required as part of the Naval Facilities Engineering Services Command (NFESC) QA Program and described in the Navy Installation Restoration Laboratory Quality Assurance Guide (NFESC, 1996). Tables 3-1 and 3-2 provide a summary of the laboratory analytical methods and associated laboratory SOPs to be used during this investigation. Laboratory SOPs are included in Appendix B.

4.3.2 Waste Characterization Analyses

Waste characterization analysis requirements will be established when the waste disposal facility is identified.

4.3.3 List of Project Target Compounds and Detection Limits

A list of the laboratory target analytes and laboratory-specific target values (TVs) for organic compounds, instrument detection limits (IDLs) for metals, and reporting limits (RLs) for all applicable analytes is provided in Table 1-5 of the SWMUs 8 and 15 QAPP (TtNUS, 2004). Tables 3-1 and 3-2 of this QAPP Addendum No. 1 identify the analyses to be conducted. The analytical fractions (i.e., VOCs, PCBs) and lead correspond to the same entries in Table 1-5 of TtNUS (2004).

4.4 LABORATORY QUALITY CONTROL CHECKS

Descriptions of various data quality indicators and the types of laboratory QC samples used to measure or verify data quality indicators (DQIs) are presented in Sections 7.0 through 7.5 of SWMUs 8 and 15 QAPP (TtNUS, 2004). Requirements for laboratory QC checks are presented in Section 7.8 of TtNUS (2004). A list of the applicable non-calibration QC requirements for confirmation sample analyses is presented in Table 4-1 of this QAPP Addendum No. 1.

4.5 DATA REDUCTION, VALIDATION, ASSESSMENT, AND REPORTING

Refer to Section 8.0 of the SWMU 8 and 15 QAPP (TtNUS, 2004) for laboratory-specific requirements. Section 8.0 of TtNUS (2004) also identifies requirements for reconciling data against DQOs.

4.6 LABORATORY PERFORMANCE AND SYSTEM AUDITS

4.6.1 Confirmation Analyses

Laboratory performance and system audit requirements are presented in Section 9.2 of the SMWUs 8 and 15 QAPP (TtNUS, 2004) for confirmation analyses.

4.6.2 Waste Characterization

These requirements will be established when the waste disposal facility is identified.

4.7 LABORATORY INSTRUMENT PREVENTIVE MAINTENANCE

Proper maintenance of laboratory instruments and equipment is essential. Depending on manufacturers' recommendations, maintenance intervals are established for each instrument. All instruments will be labeled with a model number and serial number, and a maintenance logbook will be maintained for each instrument. Personnel will be alert to the maintenance status of the equipment they are using at all times.

4.7.1 Confirmation Analysis

Refer to Section 10.2 of TtNUS (2004) for specific preventive maintenance requirements related to confirmation sampling.

4.7.2 Waste Characterization

Preventive maintenance requirements for waste characterization sampling will be particular to the laboratory performing those analyses and shall be designed to ensure that laboratory equipment is operating correctly and available for conducting the required analyses within the required time frames. These requirements will be established when the waste disposal characterization requirements are established.

4.8 LABORATORY CORRECTIVE ACTION

In general, laboratory corrective actions are warranted whenever an out-of-control event or potential out-of-control event is noted. The specific corrective action taken depends on the specific analysis and the nature of the event. Generally, the following occurrences alert laboratory personnel that corrective action may be necessary:

- QC data are outside established warning or control limits
- Method blank analyses yield concentrations of target analytes greater than acceptable levels
- Undesirable trends are detected in spike percent recoveries (%Rs) or in duplicate RPDs
- There is an unexplained change in compound detection capability
- Inquiries concerning data quality are received
- Deficiencies are detected by laboratory QA staff during audits or from performance evaluation (PE) sample test results

4.8.1 Corrective Action Documentation

Specific procedures for laboratory corrective actions are specified in the associated laboratory SOPs. Any corrective action taken above the analyst level that cannot be performed immediately at the instrument will be documented.

Laboratory corrective actions must be documented and included as part of the final evidence file. The TtNUS TOM will be informed of all major corrective actions that do not bring DQO-related nonconformances into conformance with project DQOs. The TtNUS TOM will advise all levels of project management in accordance with Section 12.0 of the SWMU 8s and 15 QAPP (TtNUS, 2004).

4.9.2 Corrective Action During Data Validation and Data Assessment

The need for corrective action may become apparent during data validation, interpretation, or presentation activities. Rework (i.e., resampling or reanalysis), a change in work procedures, or additional or refresher training are possible corrective actions relevant to data evaluation activities. The TOM will be responsible for approving the implementation of a corrective action and ensuring that it is documented appropriately for all deficiencies except those that are internal to the EMAC. The EMAC PM will be responsible for the latter corrective actions. Analytical data may be qualified during data validation to alert data users of the potential that particular analytical results are potentially deficient relative to expected performance standards. Such validation practices are described in Section 8.0 of the SWMUs 8 and 15 QAPP (TtNUS, 2004). When conducting data assessment for project decision making, a number of situation-dependent qualifications of data or decisions are possible. The number of possible situations or conditions precludes enumeration of all possible corrective actions; however, the approach used to identify and impose such qualifications is described in Section 8.0 of TtNUS (2004).

4.10 QUALITY ASSURANCE REPORTS TO MANAGEMENT

QA reports to management will be provided in four primary formats during the course of this investigation: data validation reports, reports summarizing accomplishments and QA/QC issues during the field investigation, project-wide progress reports, and laboratory QA reports. The report frequencies, content, preparers, and recipients are summarized in Table 12-1 of the SWMUs 8 and 15 QAPP (TtNUS, 2004). In that table, actions associated with the FOL will also apply to the EMAC PM, except for data validation, which will not be done on waste characterization samples.

Data validation reports for confirmation samples will address all major and minor laboratory noncompliances, as well as noted sample matrix effects. In the event that major problems occur with the analytical laboratory (e.g., repeated or extreme holding time exceedances or calibration noncompliances, etc.), the data validation manager will notify the TOM, QAM, PM, technical coordinator, and laboratory services coordinator. Such notifications (if necessary) are typically provided via internal memoranda and are placed in the project file. These reports contain a summary of the noncompliance, a synopsis of the impact on individual projects, and recommendations regarding corrective action and compensation adjustments. Corrective actions for major noncompliances are initiated at the program level.

The TtNUS FOL will provide the TtNUS TOM and the EMAC FOL will provide the EMAC PM with daily verbal field progress reports during the course of the sampling events. These reports will explain accomplishments, deviations from the QAPP, and upcoming activities, and will provide a QA summary.

The TOM and EMAC PM will provide a monthly progress report to the Navy that addresses the project budget, schedule, accomplishments, planned activities, and QA/QC issues and intended corrective actions.

The analytical laboratory for confirmation analyses will provide QA reports to TtNUS if QC limits are updated or if other significant plan deviations result from unanticipated circumstances. Because method detection limits (MDLs) will be included in the analytical data packages for confirmation and waste characterization samples, it is not necessary for the laboratories to include updated MDLs in their QA reports unless the updates result in MDLs that exceed cleanup levels or waste acceptance criteria applicable to this project. Cleanup levels are provided in the applicable IMWPs (TtNUS 2006a through 2006d); waste acceptance criteria will be provided by the waste disposal facility.

4.11 FINAL EVIDENCE FILES

Refer to Section 4.3 of the SWMUs 8 and 15 QAPP (TtNUS, 2004) for requirements governing the management of project records up to and including final disposition.

TABLE 4-1

NON-CALIBRATION QC SAMPLE USAGE FREQUENCIES, ACCEPTANCE LIMITS, AND CORRECTIVE ACTIONS
INTERIM MEASURES FOR SWMUs 7, 8, 13, AND 17
NSWC CRANE, CRANE, INDIANA

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Field Duplicate	One per 20 investigative samples collected.	Aqueous = NA Soil/Sediment = 70% RPD	Qualify data according to data validation requirements.
Equipment Rinsate Blank	NA	NA	NA
Source Water Blank	NA	NA	NA
Ambient Condition Blank	NA	NA	NA
Trip Blank	One per cooler containing samples shipped to laboratory for volatile organics analysis.	< RL (soil and water)	Identify source of volatiles contamination, if possible. Qualify data according to validation requirements. Qualify use of data if contamination appears to have adversely affected its usability.
Internal Standard	At least one internal standard per sample for GC/MS analyses	Retention times stable to ±30 seconds; area counts stable to within factor of 2	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols, and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Laboratory Control Sample	One per 20 environmental samples per matrix per analytical fraction	See Tables 7-1 and 7-2 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Laboratory Duplicate	One per 20 environmental samples analyzed for inorganic target analytes	See Table 7-1 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Laboratory Method Blank	One per 20 environmental samples or per preparation batch, whichever is more frequent	< RL (soil and water)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Matrix Spike(1)	One per analytical fraction per 20 environmental samples for organic target analytes	See Table 7-2 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).

TABLE 4-1

NON-CALIBRATION QC SAMPLE USAGE FREQUENCIES, ACCEPTANCE LIMITS, AND CORRECTIVE ACTIONS
INTERIM MEASURES FOR SWMUs 7, 8, 13, AND 17
NSWC CRANE, CRANE, INDIANA

QC Sample Type	Collection Frequency	Acceptance Limits	Corrective Action
Matrix Spike Duplicate(1)	One per analytical fraction per 20 environmental samples analyzed for organic target analytes	See Table 7-1 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Surrogate	At least one per sample for organic chromatographic analyses (GC, GC/MS, and HPLC)	See Table 7-2 of TtNUS (2004)	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).
Temperature Blank	One blank per sample cooler.	4 ± 2 °C	Laboratory action taken per LTL-1008. TtNUS action taken per validation protocols and Section 11.3 of SWMUs 8 and 15 QAPP (TtNUS, 2004).

1 Matrix spikes and matrix spike duplicates are not analyzed in the field, but additional sample material must be collected in the field to ensure that the laboratory has enough material for spiking and duplicate analysis.

GC/MS - Gas chromatography/mass spectrometry.
°C - Degrees Celsius.
HPCL - High-performance liquid chromatography.
NA - Not applicable.
RL - Reporting limit.
RPD Relative percent difference.

5.0 REFERENCES

NFESC (Naval Facilities Engineering Services Center), 1996. Navy Installation Restoration Laboratory Quality Assurance Guide.

TtNUS, 2004. Quality Assurance Project Plan for RCRA Facility Investigation at Building 106 Pond (SWMU 8) Roads and Grounds Area (SWMU 15) and Environmental Indicator Investigation for SWMU 18 (Load and Fill Area Buildings), SWMU 19 (Pyrotechnic Test Area), SWMU 20 (Crane Army Ammunition Activity Quality Assurance/Quality Control Test Area), and the Old Gun Tub Storage Lot, September.

TtNUS, 2005. Resource Conservation and Recovery Act Facility Investigation Report for Mine Fill B (SWMU 13) Naval Surface Warfare CenterCrane, Crane, Indiana Southern DivisionNaval Facilities Engineering Command, July.

TtNUS, 2006a. Interim Measures Work Plan for SWMU 7 - Old Rifle Range Old Pistol Range Site, Naval Surface Warfare Center Crane, Crane, Indiana, April.

TtNUS, 2006b. Interim Measures Work Plan for SWMU 8 - Building 106 Pond Naval Surface Warfare Center Crane Crane, Indiana, May.

TtNUS, 2006c. Interim Measures Work Plan for SWMU 13 - Mine Fill B Naval Surface Warfare Center Crane Crane, Indiana

TtNUS, 2006d. Interim Measures Work Plan for SWMU 17 - PCB Capacitor Burial/Pole Yard Naval Surface Warfare Center Crane Crane, Indiana, May.

APPENDIX A

FIELD STANDARD OPERATING PROCEDURES

A.1 TETRA TECH NUS, INC. STANDARD OPERATING PROCEDURES

APPENDIX A.1
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	Soil and Sediment Sampling
CTO0020-06	Sample Preservation, Packaging, and Shipping
CTO0020-07	Field-Portable X-Ray Fluorescence of soil and Sediment Samples
CTO0020-08	Analysis of Total Chlorinated Ethenes in Soil and Groundwater Using the Color-Tec Screening Method
CTO0020-09	Management of Investigation-Derived Waste
CTO0020-10	Field Analysis for PCB by Test Kit

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 CTO0020-01

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. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090	Project: Location: 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 tags
Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Confirmation Samples

All confirmation samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

3.1.1 Confirmation Sample numbering Scheme

The sample tracking number will consist of an seven-segment alpha-numeric code that identifies the sample's associated solid waste management unit (SWMU), matrix, sample type, location type, location number, a round number for aqueous samples or a depth interval for solid samples and, for aqueous samples, where applicable, whether a sample is filtered. Filtered samples are not expected to be collected for this project but this filtered/unfiltered segment is included in the sample nomenclature to accommodate that need, should it arise.

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

NN	**AA**	**AA (indicates sample type)**	**A (Location type)**	**NNN**	**NN Aqueous only**	**A (Soils and Sediment only)**
SWMU Number	Matrix code	Sample Type (C for composite; G for Grab)	F for excavation floor; W for excavation wall, T for tank outlet	Location number	Round Identifier and/or Filtered	Sequential depth interval from freshly exposed surface

IM = interim measures

Character Type:

A = Alpha
N = Numeric

SWMU Number (NN):

07 = Old Pistol Range
08 = Building 106 Pond
13 = Mine Fill B
17 = PCB Pole Yard

Matrix code (AA):

SO = Soil Sample
SD = Sediment Sample
WS = Waste Soil or Sediment
WW = Waste water

Individual aliquots used to form composite samples will be labeled as "A", "B", "C", or "D". Each of the aliquots for a given sample will be recorded in the field log. The number of aliquots combined to form a composite and the locations of the aliquots within the sampled area must be recorded in the "OBSERVATONS/NOTES" section of the Soil and Sediment Sample Log Sheet.

Sample type (AA):

C	=	Composite sample consisting of multiple aliquots blended to form a single mixture
G	=	Grab sample

Location Type (A):

The sample location code is the soil sample location, sediment sample location, or the tank sample location for water samples.

F	=	Excavation floor
T	=	Wastewater collection tank outlet
W	=	Excavation wall

Location number (NNN):

Sequential number beginning with "001" for each type of location (see Location Type).

Round Identifier and/or Filtered:

This code section will be used for aqueous samples only.

Round Identifier:

A two-digit round identifier will be used to track the number of aqueous samples taken from a particular aqueous sample location (i.e., tank outlet). The first sample collected from a location will be assigned round identifier 01, the second 02, etc. This applies to both existing and proposed monitoring wells and surface water locations.

Filtered:

Water samples that are field filtered (dissolved analysis) will be identified with an "F" in the last code section. No entry in this segment signifies an unfiltered (total) sample.

Depth Interval:

This code section will be used for soil and sediment samples only.

The depth code is used to denote sampling at a particular x-y location. Because continued excavation at a location could expose new ground surface with each lift of material removed from the excavations, single alphabetic character will be used to designate sequential samples from increasing depths below original grade. The letter "A" will represent the first sample collected from a particular x-y location, "B" will be used for the next sample taken from the same x-y location, but at a deeper depth than the "A" sample, etc. In this case, because samples are composite samples, "location" refers to a particular sampling grid represented by a composite sample. Each composite samples will be comprised of aliquots collected from the first six inches below the exposed surface. The "A". "B", etc. depth indicators should not be confused with the "A", "B", "C", and "D" labels used to identify individual aliquots associated with a particular sample.

3.1.2 Examples of Confirmation Sample Nomenclature

A composite soil sample collected from an excavation floor at SWMU 13, sampling grid 003, for PCB test kit analysis would be labeled as "13SOCF003A". The individual aliquots should be identified as Aliquots "A", "B", "C", and "D" for sample 13SOCF003A. If excavation continued in the same area to expose deeper surface, the next sample at that location would be labeled "13SOCF003B". The individual aliquots should be identified as Aliquots "A", "B", "C", and "D" for sample 13SOCF003B.

A composite soil sample collected from an excavation side wall at SWMU 07, sampling grid 006, for field XRF analysis would be labeled as "07SOCW006A". The individual aliquots should be identified as Aliquots "A", and "B" for sample 07SOCW006A. If excavation continued to expand the excavated area, the sample number for that next area would be labeled "07SOCW007A". Note that the sequential number "006" has been incremented to "007." The individual aliquots should be identified as Aliquots "A", and "B" for sample 07SOCW007A.

3.2 Waste Characterization Samples

Wastewater characterization samples will be numbered with unique identifiers.

3.2.1 Waste Characterization Sample Numbering Scheme

The numbering scheme for waste characterization samples will be the same as the scheme used for numbering Confirmation Samples, with the following exceptions:

- The matrix code will be "WS" or "WW" instead of "SS" or "SO".
- There will be no depth interval codes for aqueous waste water samples but depth intervals may be required for soil/sediment samples collected from roll-off boxes, waste piles, etc.

See Section 3.1 for details.

3.2.2 Examples of Waste Characterization Sample Numbers

The first wastewater grab sample collected for laboratory analysis from Tank 001 at the Building 106 Pond (SWMU 08) would be numbered as "08WWGT00101" for an unfiltered sample. A filtered sample from the same location would be labeled "08WWGT00101-F" if it corresponded to the first sample collected from that location. The next filtered and unfiltered samples collected from that location would be "08WWGT00102" and "07WWGT00102-F," respectively.

3.3 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the IMWP (TtNUS, 2006a through 2006d) and this QAPP Addendum No. 1. They will be designated using a different coding system than the one used for regular field samples.

3.3.1 QC Sample Numbering

The QC code will consist of a three- to four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AA	NNNNNN	NN	F
QC Type	Date	Sequence Number (per day)	Filtered (aqueous only, if needed)

The QC types are identified as:

TB = Trip Blank

FD = Field Duplicate

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

3.3.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day for a filtered wastewater water sample collected on June 3, 2000 would be designated as FD06030001F.

The third duplicate of the day taken of a soil sample collected on November 17, 2003 would be designated as FD11170303.

The first trip blank associated with samples collected on October 12, 2000 would be designated as TB10120001.

STANDARD OPERATING PROCEDURE
NUMBER CT020-03

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	TYPE OF ANALYSIS								COMMENTS
						CONTAINER TYPE PLASTIC (P) or GLASS (G)								
						PRESERVATIVE USED								

1. RELINQUISHED BY	DATE	TIME	1. RECEIVED BY	DATE	TIME
2. RELINQUISHED BY	DATE	TIME	2. RECEIVED BY	DATE	TIME
3. RELINQUISHED BY	DATE	TIME	3. RECEIVED BY	DATE	TIME
COMMENTS					

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

3/99
FORM NO. TtNUS-001

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

SOIL AND SEDIMENT SAMPLING

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedure for soil and sediment sampling to support Interim Measures at SWMUs 7, 8, 13, and 16 in accordance with the requrieemnts of this QAPP Addendum No. 1.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

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

Sediment Sample Log Forms: A copy of this form is attached at the end of this SOP.
Bound field logbook
Disposable plastic trowels
Labeled sample containers: See SOPs CTO0020-01 and CTO0020-02 for sample identification procedures. Sample containers are certified clean by the laboratory supplying the containers.
Plastic storage bags
Shipping containers (containing ice)
Surgical gloves
Indelible marker
Chain-of-Custody Form

3.0 SEDIMENT SAMPLE LOCATION SELECTION

The sampling personnel will determine specific sampling locations with the goal of providing a representative sample of the excavated areas.

4.0 SAMPLING PROCEDURES

The sampler will wear clean, disposable, surgical gloves. Clear vegetative matter or debris, if present, from the each sample location using a disposable sampling trowel or spoon.

4.1 Soil or sediment samples collected for volatile organics analysis in a fixed base laboratory will be obtained directly from soil or sediment surfaces using three EnCore samplers for each VOC sample. These samples are to be collected by pushing the EnCore samplers directly into the soil or sediment. All three EnCore sample containers will be collected as close to each other as possible. Make sure that all caps are securely fastened to the samplers and locked in place with both clips (see instructions that come with samplers). Write the sample identification on the strip labels that come with the samplers and place a label on each of the four samplers.

4.2 Place the three EnCore samplers for a particular sample in the ziplock pouches that come with the samplers and fill in appropriate information, including sample identification, date, time, and other information on the label. Place the four pouches in a plastic bag and place the tag on the bag, identifying the sample identification and other necessary information (see SOP CTO020-01).

4.3 Once the Encore samples are properly labeled and bagged, place the sample into the cooler containing ice and a trip blank. The cooler should be kept at 4°C and shipped to the analytical laboratory for preservation or extraction within 48 hours.

4.4 Fill in the required information on the Soil Sample Log Sheet (attached at the end of this SOP) and fill in the required information on the Chain-of-Custody (COC) Form.

4.5 For samples requiring non-VOC analyses use the trowel to dig up and homogenize the soil or sediment in an 10-inch-diameter circular area that is 6 inches deep at each aliquot location. Note the location of each aliquot in the "OBSERVATIONS/NOTES" section of the Soil and Sediment Sample Log Sheet. Stir the soil or sediment within the circular area; do not move the soil or sediment outside the circle. Also, do not dig or stir soil or sediment that is deeper than 6 inches below the ground surface.

4.6 Combine all required aliquots in the bowl in approximately equal proportions by volume. Mix the material to a uniform consistency or until the consistency does not change visibly. Use the same trowel to scoop the homogenized soil or sediment into the requisite labeled sample containers for field analysis. All sample containers will be filled in the following sequence:

- Non-VOC organics
- Inorganics (e.g., lead)

4.7 Record the sample time (using military time) on the Soil and Sediment Sample Log sheet and sample container labels. Record all other required information on the labels as specified by SOP CTO0020-01.

4.8 For field analysis, remove a portion of the sample from the container. Retain the remainder of the sample for submittal for laboratory analysis, as appropriate.

4.9 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.

4.10 Record the date, sampling site, site conditions, location map, and other information on the Soil and Sediment Sample Log Sheet. Enter the sample information onto the Chain-of-Custody Form in accordance with SOP CTO0020-03.

5.0 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

OBSERVATIONS / NOTES:

MAP:

Circle if Applicable:

MS/MSD	Duplicate ID No.:

Signature(s):

STANDARD OPERATING PROCEDURE
NUMBER CTO020-06

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

FIELD PORTABLE X-RAY FLUORESCENCE ANALYSIS OF SOIL AND SEDIMENT SAMPLES USING THE INNOV-X ALPHA SERIES INSTRUMENT

1.0 PURPOSE

This procedure is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This method is a screening method to be used with confirmatory lead analysis using United States Environmental Protection Agency (USEPA)-approved methods.

2.0 SCOPE, APPLICATION, AND LIMITATIONS

2.1 Scope of Procedure

Analysis of any other elements beside lead using FPXRF may require changes to this Standard Operating Procedure (SOP), and are therefore outside the scope of this SOP.

Although it is possible to use FPXRF to measure analytes in situ, this SOP requires removal of a soil or sediment sample from its native environment prior to analysis. By removing, drying, and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

2.2 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of the Innov-X alpha series XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis. This training may be obtained directly from INNOV-X, an INNOV-X instrument distributor or lessor, or another trained Tetra Tech NUS, Inc. person.

3.0 ACRONYMS AND ABBREVIATIONS

FPXRF: Field portable x-ray fluorescence.

mg/kg: milligrams per kilogram.

MDL: Method detection limit.

PQL: Practical quantitation limit.

QC: Quality control.

RPD: Relative percent difference.

USGS: United States Geological Survey.

XRF: X-ray fluorescence.

4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis including equipment maintenance. Also responsible for maintaining chain-of-custody of samples after receipt from sampling personnel.

5.0 PROCEDURES

5.1 Safety

5.1.1 Radiation Safety

Radiation safety practices for the INNOV-X instrument can be found in the operator's manual. Protective shielding should never be removed by the analyst or any personnel other than the manufacturer.

An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.1.2 Protective Equipment

Analysts must wear disposable plastic gloves whenever sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

5.2 Apparatus and Materials

Apparatus and materials consist of the following:

INNOV-X Alpha Series FPXRF spectrometer with data processing unit (iPAQ) pocket personnel computer):

INNOV-X Alpha Series XRF instrument manual to match the INNOV-X Alpha Series instrument.

Aluminum drying pans or aluminum foil: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other Standard reference material (SRM) that contains lead in a concentrations range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs can be obtained from the NIST, the U.S. Geological Survey (USGS), the Canadian National Research Council, and the national bureau of standards in foreign nations. Pertinent NIST SRMs for FPXRF analysis include 2704, Buffalo River Sediment; 2709, San Joaquin Soil; and 2710 and 2711, Montana Soil. These SRMs contain soil or sediment from actual sites that has been analyzed using independent inorganic analytical methods by many different laboratories. Acceptable limits for SRM percent recoveries are usually provided with the SRM. In their absence, a limit of ± 30 percent will be used as a guideline.

Instrument Blank: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Battery charger.

Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (µm) thick.

Sample containers: glass or plastic to store samples.

Sieves: 60-mesh Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

Trowels: for collecting soil samples.

Plastic bags: used for collection and homogenization of soil samples. May also be used as sample presentation device.

Drying oven: standard convection or toaster oven, for soil samples that require drying.

Rolling pin (optional): Wooden rolling pin for crushing samples.

5.3 Sample Collection, Preservation, and Handling

Samples shall be provided to the FPXRF analyst in plastic bags. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples shall be handled in accordance with sample handling SOPs in effect for the field event.

5.4 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

5.5 Instrument Start-Up

5.5.1 Ensure the pocket PC (iPAQ) is plugged into the FPXRF instrument body and install a fully charged battery into the instrument.

5.5.2 Press the ON/OFF button on the base of the pistol grip of the instrument. If the iPAQ does not automatically power up, press the Power button in the to right corner of the iPAQ.

5.5.3 Tap the Microsoft icon at the upper left corner of the iPAQ.

5.5.4 Chose START.

5.5.5 Tap "Soil Mode" on the menu or choose Mode (bottom of screen) and then choose Soil Mode from the drop down menu.

5.5.6 Allow the instrument to warm up (approximately 3 minutes).

5.5.7 Release the manual trigger lock.

5.5.8 Standardize the instrument in accordance with Section 5.6.

5.6 Standardization/Calibration Check

It is not possible to start an analysis if the instrument has not been standardized. To verify proper calibration of the instrument it is necessary to periodically standardize it using the automated standardization procedure. This must be done anytime the instrument is restarted and every 4 hours of operation, although re-standardization may be done at any other time (e.g., when instrument drift is suspected).

5.6.1 Click the standardization piece (supplied with the instrument) on the front of the instrument, verifying that the solid portion of the standard completely covers the analysis window.

5.6.2 Select "Tap here to Standardize" or select *File → Standardize*. The red light on top of the instrument will blink indicating that the instrument is producing x-rays and the shutter is open. The amber light on the rear of the instrument will also be illuminated and a status bar will appear to display the progress of the standardization.

5.6.3 Upon successful standardization the message "Successful Standardization" will appear along with the instrument resolution. In this case tap "ok" to dismiss the completion message. If problems are encountered, either follow the prompts that appear and/or repeat the standardization. Contact the FOL if problems persist. Take note of any error messages that appear as they may be useful if the instrument manufacturer must be contacted. Additional assistance is also available in the manufacturer's instrument manual.

5.7 Quality Control

The quality control (QC) program includes analysis of blanks calibration verification checks, duplicate analyses, and field duplicate samples. For all the above areas, any identified problems and corrective

action must be documented in the instrument run log, analysis narrative report, and instrument maintenance log or standards log (as applicable). Identical operating conditions will be used for each sample.

5.7.1 Laboratory Blanks

Two types of blank samples shall be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.7.1.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected, analyze an instrument blank to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL), see Section 5.9.3) check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings, "zero" the instrument according to manufacturer's instructions.

5.7.1.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.9.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

5.7.2 Calibration Verification Checks

5.7.2.1 After performing each blank check (Section 5.7.2), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest.

5.7.2.2 If the measured lead percent recovery (See Section 5.9.1) is less than 60 percent or greater than 135 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be recalibrated, or restandardized according to the manufacture instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

5.7.3 XRF Duplicate Samples

XRF duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

5.7.3.1 Analyze an XRF duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.

5.7.3.2 If the computed RPD (See Section 5.9.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. This assessment may be aided by repeating the analysis of a sample that was analyzed previously. If the observed imprecision is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses. If this does not correct the problem notify the FOL.

5.8 Sample Analysis

Note:

This section provides sample analysis instructions, assuming that appropriate instrument start-up and calibration checks have been completed. The longer the instrument count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software. Because the XRF data will be used in a screening capacity to make preliminary decisions concerning the soil concentrations relative to 400 mg/kg, it is not necessary to obtain a high degree of accuracy or precision with the instrument. Therefore, count times should be limited to less than 180 seconds unless an usually high degree of precision is expected. To change the count time, select Options → Setup Testing and enter the same value (in seconds) to minimum and maximum count times.

Note:

Section 5.7 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

5.8.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.6, 5.7.1, and 5.7.2. Count times shall be at least 60 seconds but generally less than 180 seconds.

5.8.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.

5.8.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded in a plastic bag. Mixing shall continue for at least two minutes to ensure that the sample is well mixed. To aid mixing, the sample may be placed into a thick-walled (3 mil or thicker) gallon-sized freezer bag (e.g., Zip-Loc) and rolled flat with a rolling pin to break up large chunks of dirt.

5.8.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container (e.g., aluminum drying pan) for drying.

5.8.5 Dry the homogenized sample from Step 5.9.4 for approximately 2 to 4 hours in the toaster oven at a temperature not greater than 150°C (a setting of approximately 300 F).

5.8.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing shall continue for at least one minute.

5.8.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or place it in a thin-walled (1 mil or thinner) plastic sandwich bag (e.g., Zip-Loc).

5.8.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.

5.8.9 Present the sample to the instrument in Soil Mode.

5.8.10 Perform a single pull of the trigger to start the count. The count time shall be the same as was used for the calibrations, calibration checks, and blank analyses. The message "Test in progress" will appear on the instrument and the red light on top of the instrument and will illuminate.

5.8.11 When the predetermined count period has expired the message "Test complete" will appear on the instrument. A slight delay may also be incurred during which time the message "calculating" may appear to indicate that results are being computed.

5.8.12 Record the displayed results for lead concentration in mg/kg and the associated error on Figure 1.

CAUTION

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

5.8.13 Remix the sample in the plastic bag (or rotate the sample cup approximately one-third of a turn) then acquire another measurement by repeating Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1.

5.8.14 Repeat Steps 5.8.10 and 5.8.11. Record the result and associated error on Figure 1 (XRF Field Form).

5.8.15 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination shall be based on professional judgment of the FPXRF analyst and shall consider the degree of precision observed during calibration checks and previous sample analyses. The objective will be to ensure that the average reading reported for each sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst shall correct the analytical system or notify the FOL prior to continuing with analyses.

5.8.16 Ensure that measured results are reported to the following standards

- Results < 1000 mg/kg (or parts per million) are reported to two significant figures and results > 1000 mg/Kg are reported to three significant figures.
- All values < MDL shall be reported as the MDL and flagged with the letter "U".
- All values > MDL and < PQL shall be reported as is and flagged with the letter "B".

5.9 Calculations

5.9.1 Percent Recovery: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} X\ 100\ \%$$

5.9.2 Relative Percent Difference: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$RPD = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5\,(\text{Amount in Sample 1} + \text{Amount in Sample 2})} X\ 100\ \%$$

5.9.3 Method Detection Limit (MDL): Because the analyses governed by this SOP are semi-quantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 20 mg/kg. Care will taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 20 mg/kg will be reported as an MDL.

5.9.4 Practical Quantitation Limit (PQL): Multiply the MDL by 3 to obtain the PQL: PQL = MDL*3

6.0 REFERENCES

Innov-X Systems, Inc. Innov-X Systems X-Ray Fluorescence Spectrometers Instruction Manual. Woburn, MA. June 2002.

Stephen Shefsky, NITON Corporation. Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis of Heavy Metals In Soil. Presented at the International Symposium of Field Screening Methods for Hazardous Wastes and Toxic Chemicals. Las Vegas, Nevada. January 29-31, 1997.

USEPA (U.S. Environmental Protection Agency), Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Office of Solid Waste, Washington, D.C. January 1998.

USEPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

FIGURE 1

FIELD PORTABLE XRF SAMPLE ANALYSIS LOG SHEET

Page___ of ___

Project Site Name: ______________________ Run Length/Duration: ______________________
Project No.: ______________________ Measurement Units: ______________________
Analyzed By: ______________________ Reference/Calibration: ______________________

SAMPLING AND FIELD PORTABLE XRF RESULTS DATA

SAMPLE ID	ANALYSIS DATE	ANALYSIS TIME	ELEMENT ANALYSIS	RESULT RUN 1	RESULT RUN 2	RESULT RUN 3	RESULTS AVERAGE	ERROR RUN 1	ERROR RUN 2	ERROR RUN 3	ERROR AVERAGE

STANDARD OPERATING PROCEDURE
NUMBER SOP CTO020-08

ANALYSIS OF TOTAL CHLORINATED ETHENES IN SOIL AND GROUNDWATER USING THE COLOR-TEC SCREENING METHOD

1.0 PURPOSE

This SOP describes a field screening method for determining total chlorinated ethene concentrations in the headspace above soil and groundwater samples. The method employs colorimetric gas detector tubes to analyze the headspace gases using the Color-Tec apparatus (see Figure 1, excerpted from Color-Tec Method Standard operating Procedures Manual, Ecology and the Environment, Inc.)

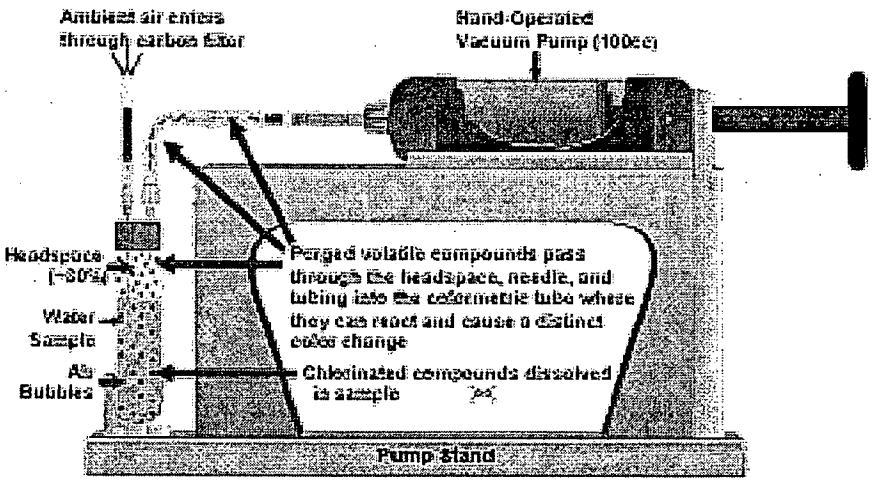


Figure 1. Color-Tec Apparatus.

2.0 DESCRIPTION

To conduct the method, groundwater or a soil and water mixture are placed in a standard 40 mL VOA vial and capped. The sample vial is then agitated vigorously for 20 seconds. Using a manual vacuum pump, two hollow needles, and Tygon® tubing, the headspace vapor is purged from the vial through the colorimetric gas detector tube. The tips of the needles are positioned so that one is in the headspace area and one is submerged at the bottom of the vial. As air is drawn from the headspace through one

needle, ambient air is allowed into the bottom of the vial through the other needle. This process purges the VOCs from the water so no headspace calculation using Henry's Constant is required.

Each colorimetric tube contains a catalyst that decomposes the chlorinated ethene, releasing hydrogen chloride, which discolors the reagent (4-phenylazodiphenylamine) in the tube. Any color change within the detector tube indicates the presence of a chlorinated ethene. The detector tubes are constructed of glass and printed with calibration scales to facilitate measurement of the linear extent of the reaction within the tube. The manufacturer of the tubes (Gastec®) provides tubes for a variety of concentration ranges. The lowest concentration range tube is used initially to screen the sample. When a positive result is observed, the concentration level is obtained by matching the linear extent of the discolored reagent inside the tube to the calibration scale printed on the outside of the tube. If the calibrated range of the tube is exceeded by the reaction, a tube with a higher concentration range is used to screen a duplicate sample. This procedure is repeated until the approximate concentration is determined.

3.0 LIMITATIONS

The color change indicated by a sample containing less than 10 µg/L PCE or less than 30 µg/L vinyl chloride is very subtle and could be missed by an inexperienced operator. The solution to this problem is practice and careful examination of each tube after pumping.

Introduction of water vapor into the tube past the catalyst stage could indicate a low level false positive to an inexperienced operator. However, water-induced false positives are easily recognized by an experienced operator and are avoidable through proper placement of the extraction needle in the VOA vial.

If chlorinated tap water is used in the soil sample screening procedure, the free chlorine in the tap water could (theoretically) indicate a low-level false positive in the soil sample. The potential for producing a false reading in this manner can be avoided by using ultrapure deionized water during the soil sample screening procedure.

Do not use hydrochloric acid as a preservative when using this method.

4.0 SAFETY PRECAUTIONS

Safety glasses should be worn whenever handling potentially contaminated soil or water.

5.0 REQUIRED EQUIPMENT

- Gastec® hand pump or equivalent
- Extra low, low, medium, and high range Gastec® colorimetric tubes
- Plastic male luer lock threaded fitting with ⅛-in hose barb
- Plastic male luer non-threaded fitting with ⅛-in hose barb
- Tygon® lab tubing ⅛-in ID x ¾-inch OD
- Deflected noncoring septum penetration needle (18G x 4)
- Hot plate
- Electrical source for hot plate
- Test tube rack
- Vortex mixer
- Charcoal prefilter tubes
- 40 mL clear VOA with Teflon-silicon septa Kimwipes™
- For soil samples, 20 mL ultra-pure deionized water per sample Chlorinated ethene QC
- standard Decontamination materials

6.0 PROCEDURE

6.1 Aqueous Samples

6.1.1 Fill 40 mL VOA vial with 24 mL (about 60 percent of volume) of groundwater sample and cap.

6.1.2 Shake the sample vigorously for 20 seconds.

6.1.3 Penetrate the septum with a needle equipped with the charcoal pre-filter tube and position the needle point near the bottom of the vial.

6.1.4 Penetrate the septum with the extraction needle attached to the Tygon® tubing. Ensure that the needle tip is well clear of the water surface.

6.1.5 Break one end of the detection tube and attach it to the Tygon® tubing.

6.1.6 Break the other end of the detection tube and attach it to the hand pump.

6.1.7 Begin drawing air from the vial and observe any change in color in the tube.

6.1.8 Record the concentration reached by the change in color on the scale marked on the tube.

6.2 Soil Samples

6.2.1 Fill 40-mL VOA vial with approximately 10 cc of soil and 10 mL of ultrapure deionized water (about 50-70 percent of volume) and cap.

6.2.2 Shake the sample vigorously for 20 seconds.

6.2.3 Penetrate the septum with a needle equipped with a charcoal pre-filter tube and position the needle point near the top of the soil.

6.2.4 Penetrate the septum with a needle attached to the Tygon® tubing. Ensure that the needle tip is well clear of the water surface.

6.2.5 Break one end of the detection tube and attach it to the Tygon® tubing.

6.2.6 Break the other end of the detection tube and attach it to the hand pump.

6.2.7 Begin drawing air from the vial and observe any change in color in the tube.

6.2.8 Record the concentration reached by the change in color on the scale marked on the tube.

7.0 QUANTITATION LIMITS

The practical quantitation limit for PCE is 5-10 ppb as measured in the headspace. This method does not employ Henry's Constant or other partitioning methods to back calculate the actual concentrations in the soil or water sample. Samples containing only trans-1,2-dichloroethene, 1,1-dichloroethene, or vinyl chloride are generally not detectable with ColorTec at concentrations below 25 µg/L.

8.0 GENERAL INTERFERENCES

Other compounds including bromine, free chlorine, and hydrogen chloride can also indicate a positive reaction within the detector tube.

The Gastec® tubes are sold to detect specific chlorinated alkenes. However, if there are other chlorinated ethenes present in a sample, the identification of a specific chlorinated compound is not possible using

the ColorTec method. The developers of the ColorTec method recommend using the Gastec® tetrachloroethene tubes for all sampling.

The detector tubes are designed to be used at temperatures of 0 to 40°C (32 to 104°F) and are calibrated based on a tube temperature (not sample temperature) of 20°C (68°F). There is a significant reduction in sensitivity of the tubes when sustained ambient air temperatures are significantly lower than 20°C while conducting the method. The reduction in the temperature of the reagent decreases the reaction time resulting in a less visible color change within the tube. Conversely, when ambient temperatures are in excess of 20°C, the tubes may be hypersensitive. The following correction factors provided by Gastec® can be applied to the readings to correct for sustained ambient temperatures other than 20°C or 68°F: Because tube/sample temperatures below 20°C significantly reduce the sensitivity of the method, it is recomended that the prepared sample vials and colorimetric tubes (prior to breaking the tips) be heated using a hot water bath prior to screening when ambient temperatures are below 20°C.

TABLE 1. CORRECTION FACTORS.

Temperature	0°C (32°F)	10°C (50°F)	20°C (68°F)	30°C (86°F)	40°C (104°F)
Correction Factor	x 2	x 1.3	x 1	x 0.7	x 0.55

SOP CTO0020-10 - Field Analysis for PCB by Test Kit

Will be provided at a later date

APPENDIX A.2

D&T ENVIRONMENTAL, LLC STANDARD OPERATING PRACTICES

APPENDIX A.2

FIELD SOP TABLE OF CONTENTS

TNFD002A	Field Records and Documentation
TNFD006B	Sampling of Surface Soils and Other Surficial Materials
TNFD010C	Sample Labeling, Control and Shipping

STANDARD OPERATING PRACTICE TNFLD002A

Field Records and Documentation

TABLE OF CONTENTS

Page

1.0 PROCEDURE 2

1.1 Materials 2
1.2 General Requirements for Field Logbooks 2
1.3 Format for Field Logbook Entries 3
1.4 What to Record in the Field Logbook 4
1.5 Site Safety and Health Logbook 5
1.6 Field Forms 5
1.7 Tracking of Field Records 6
1.8 Filing of Field Records 6

2.0 DAILY QUALITY CONTROL AND PRODUCTION REPORTS 7

STANDARD OPERATING PRACTICE TNFLD002A

Field Records and Documentation

The objectives of this Standard Operating Practice (SOP) are to provide consistent procedures and formats by which field records will be kept and activities documented, and a methodology by which field records will be managed.

1.0 PROCEDURE

1.1 MATERIALS

Materials to be used include:

- Field logbooks,
- Pens, containing indelible waterproof black ink,
- Field forms, and
- Watch with team-synchronized time (using the military 2400-hour format).

Bound, water-resistant field logbooks will only be used for the maintenance of field records (except for site specific reporting forms when required). The Project Manager prior to use must approve utilization of any other field logs.

1.2 GENERAL REQUIREMENTS FOR FIELD LOGBOOKS

Logbooks will be assigned by activity for the project until the specific activity is completed. The logbook for a specific activity will be used until the logbook is full. If the logbook for a specific activity is not filled completely, the logbook for that specific activity will be used for any future work of a similar nature e.g. quarterly groundwater sampling.

Each page of the logbook will be signed at the bottom, once filled or once field activities are completed. The logbooks will be turned in for copying/filing/tracking to the Project Manager.

The center of the front cover will be labeled with the project name and logbook number. The logbooks for each project will be sequentially numbered starting with the number one. Below the logbook number will be the activity that has been assigned and the date assigned.

All entries will be recorded in indelible, waterproof black ink. If errors are made in any field logbook, field record (form), chain-of-custody record, or any other field record document, corrections will be made by crossing a single line through the error, entering the correct information, initialing, and dating the correction.

A number of field forms have been adopted to facilitate the collection of consistent data. A reference to each specific form used should be noted in the logbook.

Sufficient information will be recorded in the logbooks and on the field forms to permit reconstruction of all field activities conducted. Duplication of information may occur between the logbooks and field forms.

1.3 Format for Field Logbook Entries

Entries in field logbooks will be made in the following format:

The date will be placed in the top right-hand corner of every right page. The time of entry recordings will be in columnar form down the left-hand side of each page. All entries should be dated and the time of entry recorded.

The first two entries in the logbook for each day will record "Personnel, Contractors on Site" and weather conditions. The time of entry onto the site and exit from the site of project personnel, subcontractors, site visitors, deliveries, and pickups will be recorded. Subcontractor problems, corrective actions, down-time (beginning and end, reason), requested field changes or variances, and approvals of requested field changes or variances will be recorded.

At the end of each day's entry or then end of a particular field event or activity, draw a downward left-to-right diagonal line from the conclusion of the entry to the bottom of the page. Sign or initial the line indicating the conclusion of the entry or the day's activity. At the end of the day's entry, record the time of departure from the work site and how the site was secured.

Language should be objective, clear, factual, and free of personal feelings or other terminology that might prove inappropriate, since field records are the basis for later written reports. Field logbooks are legal documents that must be maintained as part of the project files. The TN&A Project Manager should conduct periodic audits of field logbooks to ensure compliance with this SOP.

If not recorded on field forms, all aspects of sample collection and handling, as well as visual observations, will be documented in the field logbooks. All sample collection equipment, field analytical equipment, and equipment used to make physical measurements will be identified in the field logbooks. All calculations, results, and calibration data for field sampling, field analytical, field screening, and field physical measurements will also be recorded in the field logbooks, except where these are referenced as being recorded on approved field forms. All field analyses and measurements must be traceable to the specific piece of field equipment utilized and to the field investigator collecting the sample, making the measurement, or conducting analyses.

1.4 What to Record in the Field Logbook

Each field crew will be issued a field logbook in which detailed descriptions of all activities completed by the crew for each working day will be noted. The field logbooks will contain, at a minimum, the following:

- Name and title of the originator,
- Purpose of the field activity,
- Name, company name, and address of the site contact,
- Names and responsibilities of field crew members,
- Names and company name of all subcontract personnel,
- Names, titles, and association of any site visitors,
- Description of type and manufacturer of all heavy equipment used during field activities,
- Conversations with client representatives, senior TN&A personnel, landowners, regulatory agency representatives, etc.,
- Sample collection method,
- Quantity, location, and volume of sample(s) collected,
- Location, description, and log of photographs,
- References for all maps of the sampling site(s),
- Information regarding sampling changes, scheduling modifications and change orders,
- Information regarding drilling decisions (also recorded on the boring log),
- Information regarding sampling decisions,
- Information regarding access agreements, if applicable,
- Details of the sampling location, including a sketch map illustrating the sampling location (if not on the boring log),
- Equipment used,
- Date and time of sample collection and name of collector,
- Sample identification number(s),
- Information from containers, labels of reagents used, deionized and organic-free water used, etc.,
- Field analyses, conducted by whom, using what instruments or test kits,
- Field observations,
- All calculations and results,
- Extraordinary occurrences,
- Types of field instruments used and purpose of use, including calibration methods and results (if not on instrument calibration log),
- Investigative Derived Waste (IDW) documentation information including:
    - types of containers/drums,
    - contents, type, and approximate volume of waste,
    - type of contamination and predicted level of contamination based on available information,
- Shipping information, and
- Diagonal line with signature at end of logbook entry.

The initial four pages of the logbook will be reserved for compiling a "List of Contents" for the logbook. Information recorded will include the project name and number, duration of involvement, brief description of activities, the page numbers in the logbook, the last page number copied to file, and date copied. The personnel assigned the logbook should update the "List of Contents" on a weekly basis.

1.5 Site Safety and Health Logbook

A separate Site Safety and Health (SSH) logbook may be maintained by the Site Safety and Health Officer (SSHO) to document all daily safety and health activities. If a separate SSH logbook is not required for a project, then the health and safety information should be recorded in the field logbook. The SSHO will be responsible for ensuring the following information is entered into the logbook:

- When the daily health and safety meeting/briefing was conducted (use daily form to record meeting topics and attendees),
- Daily health and safety site inspections,
- Daily inspection of field equipment,
- Weather, including general weather conditions, temperature, wind and direction,
- Major changes in weather conditions,
- Problems associated with field monitoring equipment,
- Detailed record of any health and safety incidents at the site, including any subcontractor incidents,
- Medical problems with any field team member, including anyone taking special medications, and
- Any visitors introduced at the site along with health and/or training records, as appropriate.

1.6 Field Forms

As appropriate, the field forms listed below will be distributed by the Field Team Leader (FTL) or designate team member to field personnel. The Field Team Leader will ensure that the forms are filled out correctly by the field team members:

FORMS:

Drilling Log

Well Construction Log

Well Development Log

Well/Boring Abandonment Log

Groundwater Sampling Log

Groundwater Level Measurement Log

Sampling Logs (by media or as designated in site-specific Sampling and Analysis Plan)

Decontamination Form

Chain-of-Custody Form

Field Equipment/Meter Calibration Form

Site Safety Health Plan (SSHP) Forms:

Air Monitoring Log

Daily Site Safety Briefing Form

Contractor SSHP Acceptance Form

Employee SSHP Acceptance Form

Visitor Sign-In

Project-specific forms should be listed in the project work plan or sampling and analysis plan.

1.7 Tracking of Field Records

Field records (logbooks and forms) tracking is the responsibility of the Field Team Leader during field operations. Following completion of the field event, the FTL will deliver the field records to the TN&A Project Manager.

The FTL shall compile the original field forms in a 3-ring notebook or secure file box during field operations. At the end of the fieldwork or phases of fieldwork, the FTL will send or deliver the field forms to the TN&A Project Manager at the project office. The on-site project personnel throughout the duration of the field program will maintain copies of the field forms.

1.8 Filing of Field Records

When an individual logbook is full or the activity is completed, the logbook will be submitted to the TN&A Project Manager within two working days for final cataloging and filing. The logbooks will be stored in the project file and will only be accessible to the Project Manager.

All non-bound field records (e.g., field forms, drilling logs) will be completed on the day the associated activity occurs and will be turned in to the FTL by the end of the day. The unbound daily field forms should be sent on at least a weekly basis to the Project Manager.

All field data collected using electronic data loggers or computer entry forms will be downloaded to a computer. If possible, the data will be downloaded on a daily basis by the person collecting the data. The Field Team Leader will be responsible for ensuring that all data collected are adequately represented in electronic media and in the project file. A hard copy of the data, and any graphical representation produced by logging software, will also be printed out and filed in the project file.

2.0 DAILY QUALITY CONTROL AND PRODUCTION REPORTS

The U.S. Army Corps of Engineers (USACE) has developed a construction quality control and production reporting protocol that includes a number of forms that are commonly used on federal government projects. The following is a list of the forms applicable to environmental and construction activities such as sampling, well installation, remedial system installation, removal actions, demolition, etc. Generally, a construction quality control plan is prepared to document quality control responsible persons, procedures, and required documentation. Some of the most typical quality control forms are listed below:

- Preparatory Phase (submitted once during planning phase)
- Initial Phase (submitted on first day of project)
- Contractor Quality Control Reports (CQC Daily Report) also called Daily Quality Control Report (DQCRs) (submitted daily during field or construction activities)
- Daily Contractor Production Report (submitted daily during field or construction activities)
- Testing Plan and Log
- QC Completion Inspection (at end of field event or construction activity)
- Variance Forms
- Rework Items List
- Field Change Request
- Log of Submittals

Confirm with the client what the forms are required and who is to receive them (project distribution list). Use established templates for each form. Any changes to the forms should be approved by the client representative ahead of time. The field team leader, construction manager, or other designated project personnel is responsible for timely submittal of forms. Generally, the project manager reviews and maintains a file of the forms.

Daily CQC Reports are completed daily by the field team leader or site manager. The purpose of these reports is to provide a daily report of construction and/or field activities to the client representative, base representative (e.g., Resident Officer in Charge of Construction), and TN&A Project Manager. Information on the CQC Reports should be in a bulleted format (concise and brief). The information will include at minimum:

- Project, contract, and delivery order/task order numbers
- Date (on each page)
- Project name and location
- Temperature range, sun/wind conditions
- Summary of site activities
- Level of health and safety protection used
- Field instruments used, calibrations performed
- Problems encountered, corrective actions taken
- Sampling or testing activities performed (record sample IDs)
- QC samples collected (record QC sample IDs)
- Additional remarks, and

- Signature of person filling out CQC Report.

CQC Reports will be completed and faxed or emailed on a daily basis to the client representative and TN&A Project Manager during field events or construction activities.

Daily Contractor Production Reports are also filled out and submitted on a daily basis along with the CQC Report. The Contractor Production Reports documents work performed, health and safety actions and inspections, equipment and personnel on site, number of hours for equipment and personnel, equipment and material received, and remarks (generally production achieved). The Production Report references specific plan documents or work plan sections for each activity.

STANDARD OPERATING PRACTICE TNFLD006B

Sampling of Surface Soils and Other Surficial Materials

TABLE OF CONTENTS

1.0 INTRODUCTION 2
1.1 Objective 2
1.2 Applicability and Limitations 2
2.0 SAMPLING EQUIPMENT 2
2.1 General Sampling Supplies 2
2.2 Soil Sampling Equipment 3
3.0 PROCEDURES 3
3.1 Preparation 3
3.2 Collection Of Samples 4
3.2.1 Collection of Surface-Soil Samples for Semi-volatile Organic Compounds or Inorganic Analyses 4
3.2.2 Collection of Surface-Soil Samples for Volatile Organic Compounds Analysis 4
3.2.3 Collection and Homogenization of Composite Samples 5
3.3 Sieving Soil Samples 6
3.3.1 Objectives of Sieving Soil Samples 6
3.3.2 Equipment and Supplies 6
3.3.3 Field Procedures 6
4.0 REFERENCES 7

STANDARD OPERATING PRACTICE TNFLD006B

Sampling of Surface Soils for Site Characterization or Risk Assessment

1.0 INTRODUCTION

1.1 OBJECTIVE

The objective of this Standard Operating Practice (SOP) is to define the techniques and requirements for collecting grab surface-soils and other surficial material samples for the purposes of site characterization and/or risk assessment.

1.2 APPLICABILITY AND LIMITATIONS

Surface soil is usually referred to as the soil extending from the ground surface to a depth of approximately 1 foot (ft) below land surface. For human health risk assessment purposes (analysis of potential soil ingestion, inhalation of particulates), surface soil samples are typically collected from the upper six inches of soil. The project Sampling and Analysis Plan (SAP) or comparable document should specify the depth at which surface soil samples are to be collected.

Surface-soil samples are collected to determine the type(s) and level(s) of contamination, to define the area affected by contaminants, and to determine background concentrations. These samples may be collected as part of an investigative plan, site-specific sampling plan, and/or as a screening method for "hot spots" that may require more extensive sampling.

This SOP may also apply to collection of samples from other types of surface materials (fills, sediments, or other relatively dry, non-cemented contaminated media). This SOP does not cover radioactive materials.

2.0 SAMPLING EQUIPMENT

2.1 GENERAL SAMPLING SUPPLIES

Equipment and supplies that are typically needed for sampling surface soils and other surficial materials include:

- site-specific SAP or sampling plan,
- field logbook and/or sampling forms and site map,
- indelible (waterproof) ink pens and markers,
- sample tags/labels and the appropriate forms/documentation (including chain-of-custody forms) as described in TN&A Standard Operating Practice (SOP) 010C,

- appropriate sample containers, insulated cooler, ice for sample preservation (if required),
- latex or nitrile gloves,
- plastic zip-top bags and waterproof sealing tape,
- plastic sheeting
- decontamination equipment (e.g., rinse bottles and pressurized spray tanks) and supplies [e.g., American Society for Testing Materials (ASTM) Type II organic-free water, Alconox®, etc.]. See TN&A SOP 011A for decontamination procedures,
- protective clothing and gear, and
- Site-specific Safety and Health Plan (SSHP).

2.2 Soil Sampling Equipment

The following list provides general guidance to field personnel preparing for a field event including surficial soil sampling:

- Appropriate equipment and field screening instruments (e.g., photoionization detectors, XRF) for obtaining field measurements as specified in the site-specific SAP,
- Distance measuring device (e.g., tape measure),
- Sampling device (e.g., soil auger, slide-hammer sampling assembly, stainless steel trowel). Note: If sampling for volatile organic compounds (VOCs), the sampling device can be Teflon®-lined or constructed of stainless steel, brass, or mild steel. When sampling for metals, the sampling device must be stainless steel or plastic.
- Stainless steel, brass, Lexan® or Teflon inserts for auger sampler (if used),
- Plastic end caps for the sleeves, aluminum foil, and Teflon or silicon tape,
- Stainless steel and/or Teflon-lined pans, trays, or bowls for compositing and mixing sample material,
- Stainless steel and/or disposable plastic scoops, shovels, trowels, spoons, or spatulas,
- Stainless steel tweezers or forceps.

3.0 PROCEDURES

3.1 Preparation

When preparing for sample collection:

1. Personnel will first don appropriate protective clothing and equipment as required by the SSHP.
2. Create a clean work surface for sample collection using plastic sheeting on the ground or a portable table near the sampling area, and place decontaminated sampling equipment, sampling containers, and cooler on it. If equipment is to be decontaminated in the field, place another plastic sheet on the ground for decontamination equipment and supplies.

This will serve as a decontamination area for sampling equipment after sampling is completed.

3. Record all information in the field logbook. Document all deviations from procedure(s) and rationale for changes in the field logbook and on additional forms, as required by the SAP.

3.2 COLLECTION OF SAMPLES

3.2.1 Collection of Surface-Soil Samples for Semi-volatile Organic Compounds or Inorganic Analyses

1. Follow the sampling pattern outlined in the site-specific SAP. When known or suspected contamination exists (e.g., spill area or dumping site), attempt to collect samples starting from the least-contaminated and ending with the most-contaminated locations.
2. Change gloves in between samples and use decontaminated equipment at each site to minimize cross-contamination.
3. Label each container with the appropriate information.
4. Carefully remove or excavate loose debris and exposed material from the top 1 to 2 centimeters (cm) or to the desired sampling depth.
5. Using a clean scoop, trowel, shovel, bucket auger, or trier, place sufficient material into a clean stainless steel or Pyrex® bowl or tray and thoroughly mix the sample with a clean spoon or spatula.
6. Fill the sample container(s) directly from the tray or bowl with the spatula or spoon; remove stones, twigs, grass, etc., from the sample by hand (gloved) or forceps.
7. If the sample is water saturated, carefully decant the water from the container(s) with minimal disturbance to the sample.
8. Immediately wipe any dirt and grit from the threads of sample containers with the gloved hand. Secure and seal the Teflon-lined cap.
9. Rinse the outside of the filled sample container(s) and wipe dry.
10. Wrap the container in bubble pack (if necessary) and seal it into zip-top or other resealable plastic bag.
11. Samples for nonvolatile organics or inorganics can also be collected using liners following the procedures described in Section 3.2.2.
12. Place the sample in the cooler and cool to 4°± 2° Celsius (C). Refer to SOP 010C for the proper shipping procedures.
13. Collect appropriate location, sample-depth, and/or field measurements and record these data in the field logbook.

3.2.2 Collection of Surface-Soil Samples for Volatile Organic Compounds Analysis

1. Follow steps 1 through 5 from Section 3.2.1.

2. Assemble a clean slide-hammer sampling assembly, hand auger, or other sampler with a stainless steel, brass, or Teflon liner. The type and material of the liner will be specified in the SAP.
3. Collect the sample by advancing the sampler into the subsurface.
4. Withdraw the sampler from the subsurface and extract the sleeve (if used).
5. If the sample is water saturated, carefully decant the water from the sleeve with minimal disturbance to the sample.
6. If using sampler liner, place Teflon or aluminum foil (shiny side away from sample) sheet on ends and cap. Secure the caps with inert tape (e.g., silicon or Teflon). Wipe clean, label, and place in re-sealable plastic bag.
7. If not using liner, immediately transfer sample material into proper containers. Add field preservative, if required. Note: Do not composite or homogenize the VOC sample.
8. Place the sample in the cooler and cool to 4°± 2°C. Refer to SOP 010C for the proper shipping procedures.
9. Collect appropriate location, sample-depth, and/or field measurements and record these data in the field logbook.
10. Backfill the sample hole and flag or stake as required.

The procedures outlined above also apply to any other specified sample that can be degraded by aeration.

3.2.3 Collection and Homogenization of Composite Samples

Composite samples consist of a series of discrete grab samples that are mixed together to characterize the average composition of a given material. The discrete samples used to make up a composite sample are of equal volume and are collected in an identical fashion. A composite sample of surface soil, sediment or other surficial material is generally an areal composite. Areal composites are composed from grab samples of equal volume collected in an identical manner. The site-specific SAP should specify the basis (random grid, targeted, etc.) for collection of the grab samples.

It is important that a composite sample be truly representative if the various sample locations making up the composite. Therefore, proper homogenization techniques should be followed to generate a composite sample. In addition, the equipment used to composite the sample must not affect the sample quality. A stainless steel bowl and stainless steel or Teflon® or PFTE spoon, properly decontaminated (SOP 011A), are typically used for field compositing of soil samples.

The following steps must be followed when compositing surface soil, sludge, and sediment samples:

1. Determine where composite sample(s) will be obtained as indicated in the site-specific SAP.

2. VOCs—and in some cases, semivolatile organic compounds—should not be composited as the sample will become aerated and will not be representative of in-situ conditions.
3. Collect a minimum of three equal-volume samples from the specified sample location. The volume of each sample must be at least the amount required for a single sample.
4. Place the samples in an appropriate mixing tray or bowl.
5. Divide the soil in the sample tray or bowl into quarters. Each quarter is mixed individually, then all quarters are mixed into the center of the pan. Follow this procedure several times until the sample is adequately mixed. If a round bowl is used, stir the material in a circular fashion and occasionally turn the material over. The extent of mixing will depend on the nature of the material and should be done to achieve a consistent physical appearance prior to filling sample containers.
6. Once mixing is completed, divide the sample material in half and fill containers by scooping sample material alternately from each half. Transfer subsamples of the composited sample into the appropriate sample containers. Seal, wipe clean, and label sample containers. Use the same care in handling these samples as that used for other samples from the site.

3.3 Sieving Soil Samples

3.3.1 Objectives of Sieving Soil Samples

Field sieving of soil samples may be performed to remove coarse-grained sample material and/or determine grain-size distribution. Soil samples must be processed through multiple sieve sizes to determine grain-size distribution, in accordance with ASTM D422.

3.3.2 Equipment and Supplies

- Stainless steel and/or Teflon-lined pans, trays, or bowls for compositing and mixing sample material,
- Stainless steel and/or disposable plastic scoops, shovels, trowels, spoons, or spatulas,
- Sampling device (e.g., soil auger, slide-hammer sampling assembly, stainless steel trowel). Note: If sampling for metals, the sampling device must be stainless steel or plastic,
- No. 10 mesh (2.0- millimeter [mm]) stainless steel sieve, and other sieve sizes as required for project
- Digital scale

3.3.3 Field Procedures

1. Calibrate and "zero" the scale
2. After collecting a sample as directed in Sections 3.1 and 3.2 .1 above, weigh the total sample and record the information in the logbook.
3. Place the sample into the sieve or sieve series and shake until the separated fraction has been produced from each sieve size. A shaker may be used.

4. Weigh each soil fraction; determine volume of each fraction if needed for determination of mass.
5. Collect desired samples from sieved fraction of the sample; place in sample container, record fraction size on label as well as sample ID. Manage samples following chain-of-custody procedures.
6. Record the weights, volumes, sample descriptions, sample processing methods and equipment in the field logbook.

4.0 REFERENCES

Addiscott, T. M., and Wagenet, J. R., "A Simple Method for Combining Soil Properties That Show Variability," *Soil Sci. Soc. of A.J.*, **49**, 1365–69, 1985.

American Society for Testing and Materials, D1452-80 (reapproved 1990) Standard Practice for Soil Investigation and Sampling by Auger Borings.

American Society for Testing and Materials, D422. Standard Practice for Soil Grain-Size Analysis.

Department of Energy, *Environmental Survey Manual*, DOE/EH-0053, October 1987.

Department of Labor, *Sampling for Surface Contamination*, Industrial Hygiene Technical Manual 680 (OSHA), pp. VIII-1, May 24, 1984.

STANDARD OPERATING PRACTICE TNFLD010C

Sample Labeling, Control and Shipping

TABLE OF CONTENTS

Page

1.0 INTRODUCTION 2

2.0 SAMPLE LABELING PROCEDURES 2

2.1 Procedures 2

3.0 SAMPLE NOMENCLATURE 3

4.0 CHAIN OF CUSTODY PROCEDURES 3

5.0 SAMPLE HANDLING PROCEDURES 5

5.1 General 5
5.2 Sample Packaging for Shipment 5
5.3 Sample Shipment Considerations (DOT Regulations) 6

6.0 REFERENCES 7

STANDARD OPERATING PRACTICE TNFLD010C

Sample Labeling, Control and Shipping

1.0 INTRODUCTION

The general objective of this SOP is to present procedures for sample identification (ID), sample control and chain-of-custody, and sample handling.

2.0 SAMPLE LABELING PROCEDURES

All sample identification, field records, and chain-of-custody records will be recorded in black waterproof, ballpoint non-erasable ink. Under no circumstances will "Sharpie" markers be used on field forms or chain-of-custody forms. If errors are made on any of these documents, personnel will cross a single line through the error and enter the correct information. All corrections shall be initialed and dated by the person performing the correction. If possible, all corrections should be made by the individual who made the error.

If information is entered using stick-on labels on sample tags, logbooks, or sample containers, subsequent removal of these labels should not be possible without leaving obvious indications of the attempt. Labels should never be placed over previously recorded information. Corrections to information recorded on stick-on labels should be made as stated in the previous paragraph.

2.1 PROCEDURES

All soil, sediment, surface water, groundwater, waste, ambient air, and vapor emission samples collected will be labeled according to the following procedures.

1. Label each sample container with the following information:
   - Date and time,
   - Sample ID number,
   - Project number ,
   - Sampler (name), and
   - Preservative .
2. Enter sample collection information in the field logbook. Begin the sample collection entry with the time at which the sample is collected (or collection is begun). Make the entry chronologically in the logbook (SOP No. 002A).
3. If no map of sampling locations is available prior to sampling, sketch a drawing of the site (not to scale) in the field logbook to provide an illustration of all sampling points. Provide

measured distances from sampling points to a fixed reference point to allow accurate placement of sample locations on figures or maps.

3.0 SAMPLE NOMENCLATURE

The sample numbering scheme used for field samples and quality control (QC) samples is defined in the project-specific Sampling and Analysis Plan, Field Sampling Plan or comparable document.

4.0 CHAIN OF CUSTODY PROCEDURES

Chain-of-custody (COC) is a continuous possession of samples from their origin to completion of analysis and archiving/disposal in the laboratory. This uninterrupted possession is required to maintain integrity of samples. The chain-of-custody record is the documentation of this uninterrupted possession of the samples. The following procedures are recommended for ensuring preservation of chain-of-custody in the field:

- To simplify the chain-of-custody record and eliminate potential litigation problems, as few people as possible should handle the samples during the investigation.
- Each field team member is responsible for the proper handling and custody of the samples collected until they are properly and formally transferred to another person or facility. If the project has a Field Sample Coordinator (FSC), the field team will deliver all samples to the FSC as soon as possible, who then assumes responsibility for completing the chain-of-custody record.
- Prior to or immediately following sample collection, sample labels shall be completed for each sample container, using black waterproof, non-erasable ink, and affixed to the sample container. Labels should be affixed to each sample container and completely covered with a clear waterproof tape to prevent the labels from separating from the sample containers or water damage to the labels.
- Sample containers should be placed in resealable plastic bags before placement on ice to prevent moisture from contacting the sample jars, which could affect the legibility of the sample container labels.
- All samples collected (date, time of collection, sample ID, location and other information as necessary) must be documented in bound field logbook at the time of sample collection.

To complete the chain-of-custody and to maintain an accurate record of sample collection, transport, analysis and disposal, the following methodology will be used:

1. Samples will be accompanied by a chain-of-custody form at all times. A chain-of-custody record will be completed for all samples or materials collected. A separate chain-of-custody record will be utilized for each final destination or laboratory utilized during the inspection or investigation.

2. The chain-of-custody form will be used by personnel responsible for ensuring the integrity of samples from the time of collection until shipment to the laboratory.

3. The chain-of-custody record shall accompany all samples to the final destination. The original of the record will be placed in a sealed plastic bag taped to the inside top cover of the cooler. One copy of the record will be retained by the Field Sample Coordinator, and faxed to the Project Chemist and Project Manager. The original record will be transmitted to the Project Manager after samples are accepted by the laboratory. This copy will become a part of the project file.

4. The chain-of-custody form will be signed by each individual who has the samples in his or her possession.

5. The chain-of-custody form will be initialed in the field by the person collecting the sample, for every sample. If needed, several COC forms or COC continuation forms can be used to group samples under one COC. The continuation forms will reference the original COC number and all pages will be numbered.

6. The chain-of-custody will be completed in the field to indicate project, date, location, sampler, client, etc.

7. If the person collecting the sample does not transport the samples to the laboratory or deliver the sample containers for shipment, the first block for "Relinquished by" and "Received by" will be completed in the field by the sampler and transporter, respectively.

8. The person transporting the samples to the laboratory or delivering them for shipment will sign the record form as "Relinquished by" at the time the samples are handed off to the laboratory (which will sign the "Received by" section).

9. If the samples are transported directly to the laboratory, the chain-of-custody form will be kept in the possession of the person delivering the samples.

10. If the samples are shipped to the laboratory by commercial carrier, the chain-of-custody form will be sealed in a watertight container, taped on the inside lid, and the shipping container sealed with custody seals (two on each shipping container) and tape prior to being given to the carrier.

    a. For samples shipped by commercial carrier, the waybill or air bill will serve as an extension of the chain-of-custody record between the final field custodian and receipt in the laboratory. The sender's copy of the waybill must be stapled to the sender's copy of the chain-of-custody form and filed with the original. The waybill tracking number must be entered into the logbook. Sample sender must sign with date and time as "Relinquished by" just before sealing coolers.

    b. Upon receipt in the laboratory, the sample recipient will open the shipping containers, compare the contents with the chain-of-custody record, ensure that document control

information is accurate and complete, and sign and date the record as "Received by". Any discrepancies will be noted on the chain-of-custody form.

c. In the event of the discrepancies, the samples in question will be segregated from normal sample storage and the Project Chemist immediately notified.

d. The chain-of-custody form is completed upon receipt of the samples by the analytical service laboratory. The completed chain-of-custody form will be returned to the Project Manager and maintained in the project file.

5.0 SAMPLE HANDLING PROCEDURES

5.1 GENERAL

Upon collection, all samples will be immediately placed on ice and taken to a proper location for packing, re-icing, and shipment. At all times, sufficient ice must be in the coolers to maintain a temperature of 4° C. All void spaces in the cooler should be filled with ice. Estimate no more than two to one ratio of samples to ice, except during hot weather when a one to one ratio should be used (e.g., one pound of ice per one pound of sample). This will insure samples are stored at the proper temperature.

5.2 SAMPLE PACKAGING FOR SHIPMENT

When preparing the samples for shipment to the laboratory, the following procedures will be employed:

1. Proper packing is necessary to ensure that samples arrive at the laboratory in good condition. The following protocol will be used for packaging of samples:

    a. Only waterproof metal or equivalent strength plastic ice chests and coolers will be used. Tapes that may emit VOCs will not be used within the container.

    b. Two large garbage bags or drum liners will be placed in the cooler to prevent leakage.

    c. Approximately 2 inches of inert cushioning material (vermiculite or similar absorbent material) will be placed in the bottom of the inner drum liner.

    d. All sample containers will be placed in clear, plastic, resealable (e.g. Ziploc ™) bags. Glass containers should be wrapped in bubble wrap or cushioning material to prevent breakage.

    e. Samples will be packed properly for shipment so that the bottles will not move around and/or break during shipment.

    f. Sufficient ice to maintain 4° C during shipment will be placed beneath, around, and on top of the sample containers. Additional cushioning materials will be added to prevent movement of samples during shipment.

g. The chain-of-custody form will be placed in a resealable plastic bag and attached to the lid of the cooler with clear plastic packing tape. Laboratory name and contact, courier name(s), and other pertinent information will be recorded on the form.

h. If the cooler is equipped with a drain plug, it will be taped shut.

i. The completed shipping label will be attached to the cooler.

j. "This Side Up" arrow labels will be placed on two sides of the cooler, and "Fragile" labels will be placed on all four sides.

k. Numbered and signed custody seals will be placed on the front right and back left of each cooler. These seals will be covered with clear plastic packing tape.

l. Samples will be transported by courier in an approved, cooled shipping container, ensuring that the maximum holding times between sample collection and analysis will not be violated. In general, all samples should be shipped priority overnight.

2. The weight limit of the shipper will be observed.

3. All records pertaining to the shipment of a sample will be retained in the project file (e.g., freight bills, post office receipts, and bills of lading).

5.3 Sample Shipment Considerations (DOT Regulations)

Samples in general can be classified in to hazardous materials/dangerous goods for shipment per DOT regulations. If we believe that the sample does not contain hazardous materials above reportable quantities or above TCLP hazardous characteristics, or above listed concentrations in the 40 CFR hazardous waste table including pH, corrosivity, and ignitability criteria, we can ship them regular, i.e. not under DOT requirements.

If the shipper believes that the sample contains a hazardous substance, for example chromium, then he/she should check reportable quantity (RQ) for chromium, TCLP for chromium, and sample pH (for liquid) and make a simple estimate of actual chromium content in the sample. Then add up all the chromium per cooler and see the requirements under DOT regulations for shipment of hazardous/dangerous goods shipment.

IF YOU ARE NOT DOT-TRAINING CERTIFIED, YOU CANNOT SHIP HAZARDOUS MATERIAL/DANGEROUS GOODS. To do so risks <u>large fines against the individual who signs the shipping bill.</u>

For some chemicals and compressed gases, the quantity, limitations, shipment requirements are all given in 49 CFR 172.101, especially in the Hazardous Materials Table 172.101. <u>Field personnel cannot classify samples based on the source or origin according to these regulations</u>. The shipper is supposed to know something about actual or expected chemical concentrations of the material in the shipping container. Field personnel who are unsure about shipping anything from the field should call a qualified person in the office and have the regulations reviewed for each situation.

6.0 REFERENCES

Department of Transportation, Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples, March 1981.

United States Environmental Protection Agency Region 4, Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, May 1996.

APPENDIX A.3

TOLTEST INC. STANDARD OPERATING PROCEDURES

APPENDIX A.3

FIELD SOP TABLE OF CONTENTS

SOP 1-4	Use of Field Notebooks
SOP 8-3	Soil Sampling (Surface Soils, Hard Auger, and Grab Sampling)
SOP 8-10	Packaging and Shipment of Field Samples

TOLTEST, INC.	Procedure No:	Rev. No:	Page No:
	SOP 1-4	0	1 of 5
	Effective Date: June 1, 2001		
USE OF FIELD NOTEBOOKS	Reviewed By:		
	Approved By:		

UNCONTROLLED COPY

1.0 Purpose

The purpose of this procedure is to detail the minimum requirements for the proper use and maintenance of logbooks used during the performance of a field investigation.

2.0 Scope

This procedure applies to the following logbooks, when required to be maintained, during the performance of a field investigation:

- Site Logbook;
- Field Associate Logbook;
- Health and Safety Logbook;
- Field Equipment Logbook;
- Decontamination Logbook;
- Field Logbooks; and
- Photographs.

3.0 References

None.

4.0 Definitions

Logbook - The logbook is a bound notebook with consecutively numbered pages that cannot be removed. Upon entry of data, the logbook requires signature by the responsible individual.

5.0 Responsibilities

The site logbook is issued by the Project Manager to the Field Operations Leader or other person responsible for the direction of on-site activities (e.g., Reconnaissance Survey Team Leader, Sampling Team Leader). It is the responsibility of this person (or his designee) to keep the site logbook current

while in his possession, and return it to the Project Manager or turn it over to another field team. Following the completion of all fieldwork, the site logbook is returned to the Project Manager for inclusion in the permanent project files.

Field logbooks are issued by the Field Operations Leader to the person responsible for on-site activities. It is the responsibility of this person to keep the logbook current while in his possession and return it to the Field Operations Leader following completion of all fieldwork or when the logbook is full and a replacement logbook is needed.

6.0 Equipment

None specified.

7.0 Procedure

Logbooks are initiated at the start of the first on-site activity (e.g., initial reconnaissance survey). Entries are made each day that on-site activities take place, which involve TolTest or subcontractor personnel. A current logbook is maintained throughout the field effort for each activity.

The site logbook becomes part of the permanent project file. Because information contained in the site logbook may be admitted as evidence in cost recovery or other litigation, it is critical that this document be properly maintained. All logbooks must be secured at the end of each day.

UNCONTROLLED COPY

7.1 General

The cover of each logbook will contain the following information:

- project name;
- project number;
- Project Manager's name;
- sequential book number; and
- start date and end date.

All entries should be made in black pen. No erasures are permitted. If an incorrect entry is made, the data should be crossed out with a single strike mark so as not to be obliterated then initialed and dated. At the completion of entries by any individual, the logbook must be signed at the bottom of every page.

7.2 Site Logbook

The site logbook is a controlled document which records all major on-site activities during a field

investigation. At a minimum, the following activities or events should be recorded in the site logbook:

- arrival and departure of site visitors;
- arrival and departure of equipment;
- sample pick-up (e.g., chain-of-custody form numbers, carrier, time);
- sampling activities and sample logsheet numbers;
- start or completion of borehole, trench or monitoring well installation or sampling activities; and
- health and safety issues.

Daily entries into the logbook may contain a variety of information. At the beginning of each day the following information must be recorded:

- date;
- start time;
- weather;
- all field personnel present; and
- any visitors present.

During the day, a summary of all site activities and level of personal protection should be recorded in the logbook. The information need not duplicate that recorded in other field logbooks (e.g., sample logbook, Site Geologist's logbook, Health and Safety Officer's logbook, etc.), but should summarize the contents of these other logbooks and refer to the page locations in these logbooks for detailed information. An example of a site logbook page is shown in Attachment 1-4A.

UNCONTROLLED COPY

7.3 Field Operations Leader Logbook

The requirements for the field logbooks are the same as for the site logbook, except that the book is kept up to date in real time. In general, these books never leave the site and are sequentially numbered if more than one are used. The front of the logbook lists the project number and name, the name of the contract under which the investigation is being conducted, and the date(s) of use. A field logbook is normally used by the rig geologist or by the Field Associate to record specific details of each task. Although the field logbook contains the specific field information being collected based on a task, the number of the field logbook and page numbers used for a particular day's performance will be referenced in the site logbook to include a brief summary.

7.4 Health and Safety Logbook

The Health and Safety Logbook (HSL) is used to document protection levels, health and safety training sessions and equipment meter readings that substantiate protection levels. The HSL is also used to document any abnormal occurrences or accidents. The HSL is maintained by the Project Health and Safety Officer or his designee.

7.5 Field Equipment Logbook

The purpose of the Field Equipment Logbook (FEL) is to document the proper use, maintenance and calibration of field testing equipment. Before using field equipment, the Field Operations Leader

(supervisor) shall inspect and approve the use of the field testing equipment by initialing the appropriate page in the FEL. A calibration record shall be maintained for each instrument used on site and shall be kept in the FEL. The following items shall be tracked in the FEL:

- equipment calibration status;
- equipment decontamination status;
- equipment nonconformance; and
- equipment inspection and repair records.

The person using, maintaining or calibrating field equipment shall document his or her actions in the FEL. Entries shall contain the following:

- names and signatures of persons making entry;
- date of entry;
- adjustments made during calibration or maintenance;
- name of equipment and its identifying number;
- list or reference of procedure(s) used for calibration or maintenance; and
- standard type, concentration manufacturer, lot number and expiration date of calibration standards.

UNCONTROLLED COPY

Entries in the log shall be signed and dated by the person(s) making the entry. Every page in the log will be signed and dated by the field supervisor. This signature reflects his or her review and approval of the entry validity.

7.6 Decontamination Logbook

The decontamination logbook is used to document the proper decontamination of equipment used in the field investigation. The decontamination logbook will contain the following:

- name of person performing decontamination;
- date of entry;
- equipment shall be identified as to type and serial number (where applicable);
- decontamination procedure used;
- name, manufacturer, lot number and expiration of decontamination solutions used; and
- record of organic vapor check (after decontamination).

Entries in the log shall be signed and dated by the person making the entry. Every page of the log must

be initialed by the Project Manager indicating the validity of the entry.

7.7 Photographs

The record of photographs taken at a site for the purpose of project documentation must be recorded in the site logbook or a field logbook. When movies, slides or photographs are taken of a site or any monitoring location, they are numbered to correspond to logbook entries. The name of the photographer, date, time, site location, site description and weather conditions are entered in the logbook as the photographs are taken. A series entry may be used for rapid-sequence photographs. The photographer is not required to record the aperture settings and shutter speeds for photographs taken within the normal automatic exposure range; however, special lenses, films, filters, and other image-enhancement techniques must be noted in the logbook. Such techniques should be avoided, if possible, as they can adversely affect the admissibility of photographs as evidence. Chain-of-custody procedures depend upon the subject matter, type of film and the processing it requires. Film used for aerial photography, confidential information, or criminal investigations require chain-of-custody procedures. Adequate logbook notations and receipts may be used to account for routine film processing.

Once processed, the slides of photographic prints shall be serially numbered and labeled according to the logbook descriptions.

UNCONTROLLED COPY

8.0 Attachments

1-4A Typical Site Logbook Entry
1-4B TolTest On-Site Activity Log (Form)

ATTACHMENT 1-4A

TYPICAL SITE LOGBOOK ENTRY

START TIME: DATE:

SITE LEADER:

PERSONNEL:

DRILLER:

WEATHER: Clear, 68°F, 2-5 mph wind from SE

ACTIVITIES:

1. Steam jenny and fire hoses were set up.

2. Drilling activities at well ___ resumed. Rig geologist was _____. See Geologist's logbook Not. 1, page 29-30, for details of drilling activity. Sample No. 123-21-S4 collected; see sample logbook, page 42. Drilling activities completed at 11:50 and a 4" stainless steel well installed. See Geologist's logbook, No. 1, page 31, and well construction details for well ___.

3. Drilling Rig No. 2 steam-cleaned at decontamination pit. Then set up at location of well ___.

4. Well ___ drilled. Rig geologist was _____. See Geologist's logbook, No. 2, page __ for details of drilling activities. Sample numbers 123-22-S1, 123-22-S2, and 123-22-S3 collected; see sample logbook, pages 43, 44, and 45.

5. Well ___ was developed. Seven 55-gallon drums were filled in the flushing stage. The well was then pumped using the pitcher pump for one hour. At the end of the hour, water pumped from well was "sand-freed".

6. OEPA arrives on-site at 14:25 hrs.

7. Large dump truck arrives at 14:45 and is steam-cleaned, Backhoe and dump truck set up over test pit ____.

8. Test pit ___ dug with cuttings placed in dump truck. Rig geologist was _____. See Geologist's logbook No. 1, page 32, for details of test pit activities. Test pit subsequently filled. No samples taken for chemical analysis. Due to shallow ground-water table, filling in of test pit ___ resulted in a very soft and wet area. A mound was developed and the area roped off.

9. Express carrier picked up samples (see Sample Logbook, pages 42 through 45) at 17:50 hrs. Site activities terminated at 18:22 hours. All personnel off-site, gate locked.

UNCONTROLLED COPY

Field Associate Signature

ATTACHMENT 1-4B

UNCONTROLLED COPY

UNCONTROLLED COPY

1.0 Purpose

The purpose of this procedure is to outline the requirements for sampling surface soils, hand auger sampling, and grab sampling of soils. Soil sampling for environmental projects is normally conducted to define soil types and geological characteristics present in the subsurface and to determine the nature and extent of soil contamination. Soil samples are collected at individual sampling locations across the surface area of investigation as well as at various depths within each sample location to characterize conditions and features.

2.0 Scope

Soil sampling is potentially applicable to any hazardous waste site. A variety of sampling techniques is available for collection of soil samples. These include grab sampling, split-spoon sampling, collection of hand augured soil samples, Shelby tube sampling, and continuous coring.

Collection of surface samples may require no special sampling equipment or highly detailed procedures. Samples may be obtained on or in the vicinity of a hazardous waste site and should be representative of the nature of local surface soils or sediment.

3.0 References

- U.S. Environmental Protection Agency (EPA), 1987, *A Compendium of Superfund Field Operation Methods.*

4.0 Definitions

Auger Cuttings - soil brought to the surface by the action of the augers as they are drilled into the ground.

Hazardous Waste Sample - medium to high concentration sample (e.g., source material, sludge, leachate) requiring DOT labelling and off-site laboratory handling as a high hazard sample.

5.0 Responsibilities

5.1 Project Manager

The Project Manager is responsible for overall management of field activities and ensuring that appropriate soil sampling procedures are followed.

5.2 Site Geologist

The Site Geologist directly supervises sampling procedures, classifies soil and rock samples, and directs the packaging and sealing of soil and rock samples for shipment to an off-site laboratory. Such duties may also be performed by the on-site geotechnical engineer or Field Associate as required by an individual project.

UNCONTROLLED COPY

6.0 Equipment

The following pieces of equipment may be needed to collect samples:

- Drilling equipment - capable of collecting depth-specific samples
- Stainless steel hand auger, shovel, AMS sampling equipment or post-hole digger
- Teflon® tape
- Teflon® or stainless steel spatula or short, hand-pushed core tube
- Stainless steel bowl or Teflon® mixing bowl
- Organic vapor monitoring device (photoionization detector or organic vapor analyzer)
- Appropriate sampling containers
- Reclosable plastic bags
- Decontamination supplies as specified in TolTest SOP 3-1 and/or the project work plan or sampling plan.
- Field logbook and field sampling forms

7.0 Procedure

The soil sample collection point in the field should be within two feet horizontally of the pre-determined sample location identified in the proposal or work plan. The final location of the sampling point should be defined by surveying or measuring from previously surveyed points following procedures outlined in TolTest SOP 1-6, Site Surveying and Mapping. The accuracy of the soil sampling point location will be determined by the data quality objectives. Sample collection information should be recorded in the field logbook and in the sampling forms.

Surface/air contact may be minimized by placing the sample in an airtight container immediately after collection.

Sampling and sample preparation equipment will be decontaminated in accordance with TolTest SOP 3-3, Decontamination of Manual Sampling and Drilling Equipment, prior to and after each sample is collected unless disposable equipment is used.

If necessary or required, soil sample locations can be permanently identified using a brass surveyor's pin or equivalent permanent marker inscribed with the boring location identification number and placed in a concrete form. Depth-profile sampling must comply with the above requirements. Care must be taken to prevent cross-contamination and misidentification of samples. Vertical depth control tolerances are specified in the TolTest Quality Assurance/Quality Control Manual.

Sampling equipment should be decontaminated by following TolTest SOP 3-1, Decontamination of Ground-Water and Soil Sampling Equipment.

UNCONTROLLED COPY

7.1 Surface Soils Sampling Methods

Samples of surface soils should be collected from the ground surface to a depth of approximately six inches. Samples may be collected using a short, hand-pushed core tube or a stainless steel or polyethylene scoop or shovel. Disposable equipment is preferred, especially if the materials are heavily contaminated. Reused sampling equipment must be decontaminated between uses as describe in TolTest SOP 3-1.

7.2 Hand Augered Sampling Methods

Decontaminated equipment will be used to collect the soil sample. A hand auger consists of a sample bucket attached to the bottom of a length of steel pipe that has a crossbar (handle) at the top. A hole is drilled by turning this crossbar at the same time the operator presses the auger into the ground. The hand auger is driven to the desired depth, which is usually within a few feet of the surface.

Hand-augured samples can be collected directly from the auger bucket or from liners placed within the auger bucket. Liners can be two, four, or six inches in length. The liner for volatile organic analyses is sealed with Teflon® tape and plastic end caps, labeled and sent for analysis.

Geologic data must be maintained on the TolTest Soil Boring Log (see SOP 6-3). Hand auger buckets normally are 6 inches in length. As a result, four individual sample intervals can be obtained over a two-foot section of a boring. Samples from these intervals can be analyzed individually or composited as required by the work plan.

Soil samples that are not to be analyzed for volatile organics are placed in a stainless steel bowl and homogenized with a stainless steel spoon or Teflon® spatula. Large pebbles and cobbles should be removed from the sample. The composite samples for chemical analyses are placed in appropriate containers.

Place the samples on ice in a cooler maintained at 4° Celsius (°C).

Note the sample identification, sample location (provide sketch), sampling time, and sampling personnel in the Field Logbook.

After auguring and sampling activities are completed, the borehole will be filled using soil cuttings, granulated bentonite or concrete as specified in the work plan or proposal. The ground surface will be restored to its original condition. Sampling equipment should be decontaminated following TolTest SOP 3-1, Field Decontamination of Groundwater and Soil Sampling Equipment.

UNCONTROLLED COPY

7.3 Grab Sampling Methods

Grab samples are usually obtained from auger cuttings as they are brought to the surface by the continuous flight augers during rotary drilling activities. Careful determination of the sample's depth must be made based on the speed of the augers and visual assessment of previous cuttings.

Soil samples that are not to be analyzed for volatile organics are placed in a stainless steel bowl and homogenized with a stainless steel spoon or Teflon® spatula. Large pebbles and cobbles should be removed from the sample. The composite samples for chemical analyses are placed in appropriate containers.

Place the samples on ice in a cooler maintained at 4°C.

Note the sample identification, sample location (provide sketch), sampling time, and sampling personnel in the Field Logbook.

8.0 Attachments

None.

1.0 Purpose

The purpose of this procedure is to define the requirements necessary to properly package environmental samples and provide the necessary chain-of-custody records used in sample transfers.

2.0 Scope

This procedure applies to the packaging, shipping and documentation of samples being transferred from the field to the laboratory for analysis. Specifically, this document outlines shipping and sample documentation procedures that are in accord with the U.S. Department of Transportation (DOT). This procedure is also applicable to all samples taken from uncontrolled hazardous substance sites for analysis at laboratories away from the site; however, this procedure does not take precedence over region-specific or site-specific requirements for sample transportation and chain-of-custody documentation.

Careful packaging, shipping and documentation are necessary to insure that all samples received are undamaged and authentic. Transportation of environmental samples requires proper selection of shipping method, containerization and container seal materials.

UNCONTROLLED COPY

3.0 References

U.S. Department of Transportation, 1983. *Hazardous Materials Regulations,* 49 CFR 171- 177.

4.0 Definitions

Carrier - A person or firm engaged in the transportation of passengers or property.

Chain-of-custody record form - A printed form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to the subsequent custodian. Attachment 8-10A shows a TolTest chain-of-custody form. The chain-of-custody record form is a controlled document. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under

your custody if:

- You possess the sample.
- It is in your view after being in your physical possession.
- It was in your physical possession and then you locked it up to prevent tampering.
- You have designated and identified a secure area to store the sample.

Environmental sample - low concentration sample typically collected off site and not requiring DOT hazardous waste labelling as a high hazard sample.

Hazardous waste sample - medium or high concentration sample (e.g., source material, sludge, leachate) requiring DOT labelling and contract lab handling as a high hazard sample.

Hazardous material - A substance or material in a quantity and form which may pose an unreasonable risk to health and safety or property when transported. Defined and regulated by DOT (49 CFR 173.2) and listed in Attachment 8-10B.

Hazardous waste - Any substance listed in 40 CFR Subpart D (261.30 et seq) or 40 CFR otherwise characterized as ignitable, corrosive, reactive, or EP toxic as specified under Subpart C (261.20 et seq) that would be subject to manifest requirements specified in 40 CFR 262. Defined and regulated by the Environmental Protection Agency (EPA).

Marking - Applying the descriptive name, instructions, cautions, weight, or specification marks or combination thereof required to be placed outside containers of hazardous materials.

Packaging - The assembly of one or more containers and any other components necessary to assure compliance with the minimum packaging requirements of 49 CFR 172, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank car tanks.

Placard - Color-coded, pictorial sign depicting the hazard class symbol and name to be placed on all four sides of a vehicle transporting certain hazardous materials.

Reportable quantity (RQ) - A parenthetical note of the form "(RQ-1000/454)" following an entry in the DOT Hazardous Materials table (49 CFR 172.101) indicates the reportable quantity of the substance in pounds and kilograms. If a spill of that amount or more of the substance occurs during transit or storage, a report must be filed with DOT according to 171.15-17 concerning hazardous material incident reports. If the material spilled is a hazardous waste, a report must always be filed, regardless of the amount, and must include a copy of the manifest. If the RQ notation appears, it must be shown either immediately before or after the proper shipping name on the shipping paper (or manifest). Most shipping papers and manifests will have a column designated "HM" which may be used for this purpose.

UNCONTROLLED COPY

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

5.0 Responsibilities

5.1 Project Manager

The Project Manager is responsible for determining that samples are properly packaged and shipped and for determining that the chain-of-custody procedures are implemented from the time the samples are collected to their release to the shippers.

5.2 Field Associate

The Field Associate is responsible for implementing the packaging and shipping requirements and for initiating the chain-of-custody records until they are relinquished to another custodian, to the shipper, or to the carrier.

UNCONTROLLED COPY

6.0 Equipment

- Coder
- Teflon® and nylon strapping tape
- Vermiculite or styrofoam packaging materials
- Bubble pack
- Sampling gloves
- Poly-net
- Reclosable plastic bags
- Permanent felt tip marker
- Pen, black permanent ink
- Shipping coolers
- Ice or blue ice

7.0 Procedure

7.1 Sample Packaging and Shipping

Samples collected for shipment from a site should be classified as either environmental or hazardous material (or waste) samples. In general, environmental samples are collected off site (for example, from streams, ponds, or wells) and are not expected to be grossly contaminated with high levels of hazardous materials. On-site samples (for example, soil, water, and materials from drums or bulk storage tanks,

obviously contaminated ponds, lagoons, pools, and leachates from hazardous waste sites) are considered hazardous. A distinction must be made between the two types of samples in order to:

1. Determine the appropriate procedures for transportation of samples. If there is any doubt, a sample should be considered hazardous and shipped accordingly.

2. Protect the health and safety of laboratory personnel receiving the samples. Special precautions are used at laboratories when samples other than environmental samples are received.

7.2 Environmental Samples

7.2.1 Packaging

1. Environmental samples may be packaged following the procedures outlined in Section 7.4 for samples classified as "flammable liquids" or "flammable solids". Requirements for marking, labeling, and shipping papers do not apply. Environmental samples may also be packaged without being placed inside metal cans as required for flammable liquids or solids.

2. Place the sample container, properly identified and with a sealed lid, in a polyethylene bag and seal the bag.

3. Place the sample in a fiberboard container or cooler which has been lined with a large polyethylene bag and contains ice, blue ice packs or frozen bottled water.

4. Pack with enough noncombustible, absorbent, cushioning materials to minimize the possibility of the container breaking.

5. Seal the large polyethylene bag.

6. Seal or close the outside container.

7.2.2 Marking/Labeling

Sample containers must have a completed sample identification and the outside container must be marked "Environmental Sample." The appropriate side of the container must be marked "This End Up" and arrows placed appropriately. No DOT markings or labeling are required.

UNCONTROLLED COPY

7.2.3 Shipping Papers

No DOT shipping papers are required. However, the appropriate chain-of-custody forms must be included with the shipment. Core must be taken to obtain and list the correct laboratory address on shipping documents. In addition, the type of shipment method (overnight, second day air, regular mail) must be specified as required by the project.

7.2.4 Transportation

There are no DOT restrictions on mode of transportation.

7.3 Determination of Shipping Classification for Hazardous Material Samples

Samples not determined to be environmental samples, or samples known or expected to contain hazardous materials, must be considered hazardous material samples and transported according to the requirements listed below.

7.3.1 Known Substances

If the substance in the sample is known or can be identified, package, mark label, and ship according to the specific instructions for that material (if it is listed) in the DOT Hazardous Materials Table, 49 CFR 172.101.

The following steps are provided to help in locating a proper shipping name from the Hazardous Materials Table, 48 CFR 172.101.

1. Look first for the chemical or technical name of the material, for example, ethyl alcohol. Note that many chemicals have more than one technical name; for example, perchloroethylene (not listed in 172.101) is also called tetrachloroethylene (listed in 172.101). It may be useful to consult a chemist for all possible technical names a material can have. If your material is not listed by its technical name, then

2. Look for the chemical family name. For example, pentyl alcohol is not listed, but the chemical family name is alcohol, n.o.s. (not otherwise specified). If the chemical family name is not listed, then

3. Look for a generic name based on end use. For example, Paint, n.o.s. or Fireworks, n.o.s. If a generic name based on end use is not listed, then

4. Look for a generic family name based on end use. For example, Drugs, n.o.s. or Cosmetics, n.o.s. Finally if your material is not listed by a generic family name but you suspect or know the material is hazardous because it meets the definition of one or more hazard classes, then

UNCONTROLLED COPY

5. You will have to go to the general hazard class for a proper shipping name. For example, Flammable Liquid, n.o.s., or Oxidizer, n.o.s.

7.3.2 Unknown Substances

For samples of hazardous substances of unknown content, select the appropriate transportation category according to the DOT Hazardous Materials Classification (Attachment 8-10B), a priority system of transportation categories.

The correct shipping classification for an unknown sample is selected through a process of elimination, utilizing Attachment 8-10B. Unless known or demonstrated otherwise (through the use of radiation survey instruments), the sample is considered radioactive and appropriate shipping regulations for "radioactive materials" are followed.

If radioactive material is eliminated, the sample is considered to contain "Poison A" materials (Attachment 8-10C), the next classification on the list. DOT defines "Poison A" as extremely dangerous poisonous gases or liquids of such a nature that a very small amount of gas, or vapor of the liquid, mixed with air is dangerous to life. Most Poison A materials are gases or compressed gases and would not be found in drum-type containers. Liquid Poison A would be found only in closed containers; however, all samples taken from closed drums do not have to be shipped as Poison A, which provides for a "worst case" situation. Based upon information available, a judgement must be made whether a sample from a closed container is a Poison A.

If Poison A is eliminated as a shipment category, the next two classifications are "flammable" or "nonflammable" gases. Since very few gas samples are collected, "flammable liquid" would be the next applicable category. With the elimination of radioactive material, Poison A, flammable gas, and nonflammable gas, the sample can be classified as flammable liquid (or solid) and shipped accordingly. These procedures would also suffice for shipping any other samples classified below flammable liquids in the DOT classification table (Attachment 8-10B). For samples containing unknown materials, categories listed below flammable liquids/solids on Attachment 8-10B are generally not used because showing that these materials are not flammable liquids (or solids) requires flashpoint testing, which may be impractical and possibly dangerous at a site. Thus, unless the sample is known to consist of material listed as less hazardous than flammable liquid (or solid) on Attachment 8-10B, it is considered a flammable liquid (or solid) and shipped as such. For any hazardous material shipment, utilize the shipping checklist as a guideline to ensure that all sample-handling requirements are satisfied.

UNCONTROLLED COPY

7.4 Packaging and Shipping of Samples Classified as Flammable Liquid (or Solid)

7.4.1 Packaging

Applying the word "flammable" to a sample does not imply that it is in fact flammable. The word prescribes the class of packaging according to DOT regulations.

1. Collect the sample in the prescribed container with a nonmetallic, Teflon®-lined screw cap. To prevent leakage, fill container no more than 90% full. If an air space in the sample container would affect sample integrity, place that container within a second container to meet the 90% requirement.

2. Complete the sample label and identification tag and attach it securely to the sample container.

3. Seal the container and place in a 2-ml thick (or thicker) polyethylene bag, one sample per bag. Position identification tag so that it can be read through the bag. Seal the bag.

4. Place the sealed bag inside the metal can and cushion it with enough noncombustible, absorbent material (for example, vermiculite or diatomaceous earth) between the bottom and sides of the can and bag to prevent breakage and absorb any leakage. Pack one bag per can. Use clips, tape, or other positive means to hold can lid securely, tightly and permanently. Mark the can as indicated in Paragraph 1 of Section 7.4.2, below.

5. Place one or more metal cans (or a single 1-gallon bottle) into a strong outside container, such as a cooler or a DOT-approved fiberboard box. Surround the cans with noncombustible, absorbent cushioning material for stability during transport. Mark containers as indicated in Paragraph 2 of Section 7.4.2.

UNCONTROLLED COPY

7.4.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either-hand printed or in label form, on the metal can (or the 1-gallon bottle):

   - Laboratory name and address.
   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325".

Not otherwise specified (n.o.s.) is not used if the flammable liquid (or solid) is identified. Then the name of the specific material is listed before the category (for example, Acetone, Flammable Liquid), followed by its appropriate UN number found in the DOT hazardous materials table (49 CFR 172.101).

Place all information on the outside shipping container as on the inner can (or bottle), specifically:

- Proper shipping name.

- UN or NA number.
- Proper label(s).
- Addressee and sender.

Place the following labels on the outside container: "Cargo Aircraft Only" and "Flammable Liquid" (or "Flammable Solid"). "Dangerous When Wet" label should be used if the solid has not been exposed to a wet environment. "Laboratory Samples" and "THIS SIDE UP" or "THIS END UP" should also be marked on the top of the outside container, and upward-pointing arrows should be placed on all sides of the container.

7.4.3 Shipping Papers

1. Use abbreviations only where specified. Complete the carrier-provided bill of lading and sign the certification statement (if the carrier does not provide one then use the standard industry form, see Attachment 8-10D). Provide the following information in the order listed (one form may be used for more than one exterior container).

   - "Flammable Liquid, n.o.s. UN1993" or "Flammable Solid, n.o.s. UN1325).
   - "Limited Quantity" (or "Ltd. Qty.")
   - "Cargo Aircraft Only"
   - Net weight (wt) or net volume (vol), just before or just after "Flammable Liquid n.o.s." or "Flammable Solid, n.o.s.," by item, if more than one metal can is inside an exterior container
   - "Laboratory Samples" (if applicable)

2. Include the chain-of-custody record, properly executed in the outside container.

3. "Limited Quantity" or "Flammable Liquid, n.o.s." is limited to one pint per inner container. For "Flammable Solid, n.o.s.", the net weight of the inner container plus the sample should not exceed one pound; total package weight should not exceed 25 pounds.

7.4.4 Transportation

1. Transport unknown hazardous substance samples classified as flammable liquids by rented or common carried truck, railroad, or express overnight package services. Do not transport by any passenger-carrying air transport system, even if they have cargo-only aircraft. DOT regulations permit passenger airline company cargo only aircraft, but difficulties with most of these suggest avoiding them. Instead, ship by airline carriers that only carry cargo.

2. For transport by government-owned vehicle, including aircraft, DOT regulations do not apply. However, procedures described above, with the exception of execution of the bill of lading with certification, should still be used.

UNCONTROLLED COPY

7.5 Packaging and Shipping Of Samples Classified as Poison "A"

This packaging, marking, labeling, and shipping method provides a worst-case procedure for materials classed as Poison A (49 CFR 173.328). In the absence of reliable data that exclude the possibility of the presence of Poison A chemicals or compounds (see Attachment 8-10C), these procedures must be followed.

7.5.1 Packaging

Applying the word "poisonous" to a sample does not imply that it is, in fact, poisonous, or how poisonous. It describes the class of packaging according to DOT regulations.

UNCONTROLLED COPY

1. Collect samples in a polyethylene or glass container with an outer diameter narrower than the valve hole on a DOT specification #3A1800 or #3AA1800 metal cylinder. To prevent leakage, fill the container no more than 90% full. Seal the sample container.

2. Complete the sample label and identification tag and attach it securely to the sample container.

3. Attach a string or flexible wire to the neck of the sample container and lower it into the metal cylinder partially filled with noncombustible, absorbent cushioning material (for example, diatomaceous earth or vermiculite). Place only one container in the metal cylinder. Pack with enough absorbing material between the bottom and sides of the sample container and the metal cylinder to prevent breakage and absorb any leakage. After the cushioning material is in place, drop the end of the string into the cylinder valve hole.

4. Replace the valve, torque to 250 ft-lb (for 1-inch opening), and replace valve protector on the metal cylinder, using Teflon® tape.

5. Mark and label the cylinder as described in Paragraph 1 of Section 8.5.2.

6. Place one or more of the cylinders in a DOT-approved outside container.

7. Mark and label the outside container and complete all shipping papers as described below.

7.5.2 Marking/Labeling

1. Use abbreviations only where specified. Place the following information, either hand-printed or in label form, on the side of the cylinder or on a tag wired to the cylinder valve protector.

- "Poisonous Liquid, n.o.s." or "Poisonous Gas, n.o.s. NA9035".
- Laboratory name and address.
- DOT label "Poisonous Gas" (even if sample is liquid) on the cylinder.

2. Put all information contained on the metal cylinder on the outside container. Print "Laboratory Sample" and "Inside Packages Comply With Prescribed Specifications" on the top and/or front of the outside container. Mark "THIS SIDE UP" on top of the container and place upward-pointing arrows on all four sides.

7.5.3 Shipping Papers

1. Use abbreviations only as specified. Complete a carrier-provided bill of lading and sign the certification statement (if the carrier does not provide one, use a standard industry form, see Attachment 8-10D). Provide the following information in the order listed. One form may be used for more than one exterior container.)
   - "Poisonous Liquid, n.o.s. NA9035."
   - "Limited Quantity" (or "Ltd. Qty.").
   - Net weight (wt) or net volume (vol), just before or after "Poisonous Liquid, n.o.s.", of each cylinder, if more than one is inside the outer container.

2. Include a chain-of-custody record, properly executed, in the container or with the cylinder.

3. Accompany the shipping container to the carrier and, if required, open the outside container(s) for inspection.

UNCONTROLLED COPY

7.5.4 Transportation

Transport any unknown hazardous substance samples classified as Poison A only by ground transport or government-owned aircraft. Do not use air cargo, other common-carrier aircraft, or rented aircraft.

7.6 Transport of Investigation and Remediation Wastes

The packaging, marking, labeling, and other shipping requirements will depend on the particular waste to be transported. Examples of wastes which may be generated during the site investigations are decontamination or cleaning solutions, contaminated disposable items, test pit spoils, drilling cuttings or fluids and contaminated monitoring well discharges. Waste materials from remediation include excavation spoils, overpacked drums and discharges from drained lagoons or tanks.

In many cases, wastes generated during site investigations will be disposed of on site. These relatively small volumes of waste will be dealt with as part of the waste to be cleaned up or isolated during remediation. This avenue should be pursued, if feasible, to avoid the inconvenience of transportation and disposal which are disproportionately expensive for small volumes. If such a solution is approved,

materials should be properly bagged, drummed, covered, buried, or otherwise contained at the end of each day. Those materials which must be transported for treatment, storage, or disposal should be packaged, labeled and marked in accordance with applicable regulations.

Many wastes generated during site investigations and remediation activities will probably be adequately handled under the classification "ORM-E" (i.e., other regulated materials, type E). Types of wastes which would normally fall under this classification are contaminated disposable protective clothing and sampling equipment, spent soapy decontamination solutions and rinses, contaminated drilling cuttings or fluids and contaminated soils excavated during site investigations or remediation.

Spent solvents used for decontamination of sampling equipment (e.g., acetone or methanol) should be referenced by the actual product name. Liquids from drums or tanks should be specified as accurately as possible based on results of lab analysis or reliable records. If the liquid is known to be a solvent, organic liquid, or spent distillation bottoms, it should be referenced by its actual or generic name from the Hazardous Materials Table. In cases requiring emergency actions where the identity of a substance is not accurately know, place the substance in one of the general hazard classes in 49 CFR 173.2. The choice of class should be conservative; that is use the highest priority class based on available information as described in Section 7.3.2.

UNCONTROLLED COPY

The following steps for preparing hazardous materials for shipment were extracted from the "Hazardous Materials Transportation Guide for Shippers" published by the U.S. DOT. References are to CFR Title 49:

1. Determine the Proper Shipping Name

   The shipper must determine the proper shipping name of the materials as listed in the Hazardous Materials Table, 172.101, Column (2).

2. Determine the Hazard Class or Classes

   a. Refer to the Table, 172.101, Column (3) and locate the hazard class of the material or follow the steps described in Section 7.3.2 of this Guideline.

   b. If more than one class is shown for the proper shipping name, determine the proper class by definition.

   c. If the material has more than one hazard, classify the material based on the order of hazards in 173.2.

3. Select the Proper Identification Number

   a. Refer to the Table, 172.101, Column (3a) and select the Identification Number (ID) that corresponds to the proper shipping name and hazard class.

   b. Enter the ID Number(s) on the shipping papers and display them, as required, on packaging or placards.

4. Determine the Mode(s) of Transport to Ultimate Destination*

   a. As a shipper, you must assure yourself that the shipment complies with the various modal requirements.

   b. The modal requirements may affect the following:
      (1) Packaging
      (2) Quantity per package
      (3) Marking
      (4) Labeling
      (5) Shipping papers
      (6) Certification

   * For example, truck, rail or air.

5. Select the proper label(s) and apply as required

   Required labels are based on the hazard class of the substance to be shipped. No placards are required on vehicles transporting ORM-E substances or limited quantities of any hazardous materials (e.g., hazardous samples as discussed in Section 8.4).

   a. Refer to the Table, 172.101, Column (4) for required label(s).

   b. For details in labeling refer to:
      (1) Additional Labels, 172.402
      (2) Location of Labels, 172.406
      (3) Packaging (Mixed or Consolidated), 172.404(a) and (b)
      (4) Packages Containing Samples, 172.402(h)
      (5) Radioactive Materials, 172.403
      (6) Authorized Label Modification, 172.405

UNCONTROLLED COPY

6. Determine and Select the Proper Packaging

   a. Refer to the Table, 172.101, Column (5a) for exceptions and Column (5b) for authorized Packaging. Consider the following when selecting an authorized container: Quantity per package; Cushioning material, if required; Proper closure and reinforcement.

   b. If packaged by a prior shipper, make sure the packaging is correct and in proper condition for transportation.

7. Mark the Packaging (Including Overpacks)

   a. Apply the required marking (172.300); Proper shipping name and number, when required (172.301); Name and address of Consignee and Consignor (172306).

   b. For details and other required markings, see 172.300 through 172.338.

8. Prepare Shipping Papers

   a. The basic requirements for preparing shipping papers include: proper shipping name; hazard class; ID number; total quantity; shipper's certification.

   b. Make all entries on the shipping papers, using the information required, and in proper sequence 172.202.

   c. For additional requirements, see 172.200 through 172.205.

9. Certification

   a. Each shipper must certify, by printing (manually or mechanically) on the shipping papers, that the materials being offered for shipment are properly classified, described, packaged, marked, and labeled, and are in proper conditions for transportation according to the applicable DOT Regulations (172.204).

10. Loading, Blocking, and Bracing

    When loading hazardous materials into the transport vehicle or freight container, each package must be loaded, blocked, and braced in accordance with the requirements for the mode of transport.

    a. If the shipper loads the freight container or transport vehicle, the shipper is responsible for the proper loading, blocking, and bracing of the materials. The packages must be properly labeled as to the right side up and samples must be packed to avoid damage in case of overturning.

UNCONTROLLED COPY

b. If carrier personnel do the loading, the carrier is responsible.

11. Determine the Proper Placard(s)

Each person who offers hazardous materials for transportation must determine that the placarding requirements have been met.

a. For highway, unless the vehicle is already correctly placarded, the shipper must provide the required placard(s) and required identification number(s) (172.506).

b. For rail, if loaded by the shipper, the shipper must placard the rail car if placards are required. (172.508)

c. For air and water shipments, the shipper has the responsibility to apply the proper placards.

12. Hazardous Waste/Hazardous Substance

a. If the material is classed as a hazardous waste or hazardous substance, most of the above steps will be applicable.
b. Pertinent Environmental Protection Agency Regulations are found in the Code of Federal Regulations, Title 40, Part 262.

UNCONTROLLED COPY

7.7 Chain-of-Custody Guidelines

The term "chain-of-custody" refers to procedures which ensure that evidence presented in a court of law is what it is represented to be. The chain-of-custody procedures track the evidence from the time and place it is first obtained to the courtroom. These procedures also provide an auditable trail for the evidence as it is moved and/or passes from the custody of one individual to another. In addition, procedures for consistent and detailed records facilitate the admission of evidence under Rule 803(b) of the Federal Rules of Evidence (P.L. 93-575).

Chain-of-custody procedures, record keeping, and documentation are an important part of the overall management control of the samples. Regulatory agencies must be able to provide the chain of possession and custody of any samples that are offered for evidence, or that form the basis of analytical test results introduced as evidence. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

7.7.1 Sample Identification

The following information shall be written in the sample log book when in-situ measurements or samples for laboratory analysis are collected:

- project code;
- station number;
- location of station;
- date and time of measurement;
- samples used (if any);
- field observations (include date and time);
- level of personnel protection (if required);
- equipment used to make physical measurements and collect samples; and
- calibration data for equipment used.

Measurements and observations shall be recorded using black, waterproof ink

7.7.2 Sample Label

Samples, other than in-situ measurements, are removed and transported from the sample location to a laboratory or other location for analysis. Before removal, however, a sample is often divided into portions, depending upon the analyses to be performed. Each portion is preserved in accordance with the project work plan or proposal. Each sample container is identified by a sample label.

Sample labels are provided by the laboratory or container manufacturer. The sampler fills out the following information on the sample label:

- Project number
- Sample number - The unique sample number identifying this sample
- Date - A six-digit number indicating the month, day, and year of sample collection; e.g., 12/21/95
- Time - A four digit number indicating the 24-hour time of collection (for example: 0954 is 9:54 a.m., and 1629 is 4:29 p.m.)
- Medium - Water, Soil, Sediment, Sludge, Leachate, etc.
- Sample type - Grab or Composite
- Preservation - Type, quantity, and concentration of preservative added
- Analysis - Analyses requested as part of the investigation. Both name and method number must be listed (e.g., BTEX/8020)
- Sampled by - Name of the sampler

UNCONTROLLED COPY

- Lab # - The receiving laboratory assigns the lab # to the sample label (this number is not to be used for on-site analyses)

- Remarks - If for contract lab analysis, include the contract lab case of SAS number, and contract lab sample number from the traffic report, SAS Packing List, or Dioxin Shipment Record. Also, pertinent observations of the sampler (e.g., sequence number for sequential samples).

7.7.3 Chain-of Custody Procedures

After collection, separation, identification, and preservation, the sample is maintained under chain-of-custody procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

Field Custody Procedures:

1. Samples are collected as described in the project work plan or proposal. Care must be taken to record precisely the sample location and to ensure that the sample number on the label exactly matches those numbers on the sample log sheet and the chain-of-custody record.

2. The person undertaking the actual sampling in the field is responsible for the care and custody of the samples collected until they are properly transferred or dispatched.

3. When photographs are taken of the sampling as part of the documentation procedure, the name of the photographer, date, time, site location, and site description are entered sequentially in the site logbook as photos are taken. Once developed, the photographic prints shall be serially numbered, corresponding to the logbook descriptions.

4. Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions, i.e., a logbook notation would explain that a pencil was used to fill out the sample label because a ballpoint pen would not function in freezing weather.

7.7.4 Transfer of Custody and Shipment

Samples are accompanied by the chain-of-custody record form (Attachment 8-10A). When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory. The chain-of-custody record is filled out as follows:

1. Enter header information (project number and name, Contract Lab case No. or SAS No.). For each station number, enter date, time, composite/grab, station location, number of containers, analytical parameters.

2. Sign, date, and enter the time under "Relinquished by" entry.

UNCONTROLLED COPY

UNCONTROLLED COPY

3. Make sure that the person receiving the sample signs for the "Received by" entry, or enter the name of the carrier (e.g., UPS, Federal Express) under "Received by". Receiving laboratory will sign "Received for Laboratory by" on the lower line and enter the date and time.

4. Enter the bill-of-lading or Federal Express airbill number under "Remarks or Reason for Change of Custody", if appropriate.

5. Place the original (top, signed copy) of the chain-of-custody record form in the appropriate sample shipping package. Retain a copy with field records.

6. Shipping containers should be secured to ensure samples have not been disturbed during transport by using nylon strapping tape and EPA custody seals. The custody seals should be placed on the containers so that they cannot be opened without breaking the seal.

7. Complete other carrier-required shipping papers.

   The custody record is completed using black waterproof ink. Any corrections are made by drawing a line through and initialing and dating the change, then entering the correct information. Erasures are not permitted.

   Common carriers will usually not accept responsibility for handling chain-of-custody record forms; this necessitates packing the record in the sample container (enclosed with the other documentation is a plastic zip-lock bag). As long as custody forms are sealed inside the sample container and the custody seals are intact, commercial carriers are not required to sign off on the custody form.

   The laboratory representative who accepts the incoming sample shipment signs and dates the chain-of-custody record, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal log books and custody records throughout sample preparation and analysis.

UNCONTROLLED COPY

7.7.5 Receipt for Samples Form

Whenever samples are split with a private party or government agency, a separate Receipt for Samples Record Form (see Attachment 8-10F) is prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the party or agency shall require the signature of a representative of the appropriate party acknowledging receipt of the samples. If a representative is unavailable or refuses to sign, this is noted in the "Received by" space. When appropriate, as in the case where the representative is unavailable, the custody record should contain a statement that the samples were delivered to the designated location at the designated time. This form must be completed and a copy given to the owner, operator, or agent-in-charge even if the offer for split samples is declined. The original is retained by the Project Manager.

8.0 Attachments

8-10A Chain-of-Custody Record Form and Chain-of-Custody Seal

8-10B DOT Hazardous Materials Classification (49 CFR 1732)

8-10C DOT List of Class "A" Poison (49 CFR 172.101) and Hazardous Material Shipping Checklist

8-10D Standard Industry Certification Form

8-10E Sample Label

8-10F Receipt For Samples Form

UNCONTROLLED COPY

CHAIN-OF-CUSTODY PREPARATION

UNCONTROLLED COPY

1.0 Purpose

The purpose of this procedure is to establish a standard procedure for preparation of chain-of-custody forms.

The purpose of document control is to assure that all documents for a specific project are accounted for. Chain-of-custody procedures are an integral part of this document control. The purpose of the chain-of-custody form is to assure that the samples collected in the field are being identified properly and the original integrity of a sample is not destroyed by poor collecting and/or handling techniques.

2.0 Scope

This procedure defines the procedures to prepare and complete the chain-of-custody form.

3.0 References

- U.S. Environmental Protection Agency, 1979. *Methods for Chemical Analyses of Water and Wastes,* EPA 600/4-79-020.
- National Well Water Association, 1986. *RCRA Ground-Water Monitoring Technical Guidance Document,* NWWA/EPA Series.

4.0 Definitions

None

5.0 Responsibilities

5.1 Project Manager

The Project Manager is responsible for ensuring that field personnel know the chain-of-custody

procedures.

UNCONTROLLED COPY

5.2 Field Associate

The Field Associate is responsible for ensuring the chain-of-custody form is properly prepared.

6.0 Equipment

None

7.0 Procedure

To maintain and document sample possession, chain-of-custody procedures are followed. Using these procedures and documentation gives a reasonable assurance that 1) the sample was handled properly and/or 2) if suspected of being improperly handled, witnesses are available who can disclose the information needed to prove or disprove improper handling. Writing chain-of-custody procedures facilitates the admission of evidence under Rule 803(6) of the Federal Rules of Evidence (P.L. 93-575). Evidentiary foundation is recognized by most courts as fulfilling the requirement of sample identification which shows that the original sample:

1. Was not exchanged with another sample
2. Was not contaminated or tampered with
3. Was the one tested?
4. Was the same sample from which the analytical results are derived?

UNCONTROLLED COPY

7.1 Uses of the Chain of Custody Form

Different situations in which the chain-of-custody is used are the following:

- If the person who sampled is transferring the sample(s) to another person; or
- If the laboratory is transferring the sample(s) to another outside laboratory; or
- If the person who sampled is transferring the sample(s) to the laboratory.

7.2 Chain-of-Custody Preparation Procedure

Instructions for preparation of the TolTest, Inc. chain-of-custody form is as follows:

1. Enter the following information in the appropriate sections:
   - TolTest project number or the client's identification number
   - TolTest division or the client's name
   - Purchase order number (not required for TolTest projects)
   - TolTest project manager or the person responsible for the project

- Telephone number where the project manager can be reached
- Turnaround time required in business days (standard is 10 business days)

2. PRINT the sampler's name.

3. Obtain the sampler's signature.

4. Complete the sample information sections. For the type of sample, use the following abbreviations:
   - A = auger cuttings
   - AR = Air rotary cuttings
   - C = composite
   - G = grab
   - S = split-spoon
   - ST = Shelby tube

5. Specify the analysis required and the method number, if known.

6. Note whether preservatives were added to any sample container.

7. Indicate any remarks concerning the samples.

8. Obtain the signature of the person transferring the samples.

UNCONTROLLED COPY

8.0 Attachments

8-16A TolTest Chain-of-Custody Form

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
TABLE OF CONTENTS

SOP NUMBER	REV #	QAPP	TITLE
			LAUCKS TESTING LABORATORIES
LTL-1001	4	126	Elements of SOP and Method Formats
LTL-1002	5	126	Document Tracking and Control
LTL-1003	2	126	Chain-of-Custody and Documentation Procedures
LTL-1004	4	126	Documentation of Analyst Competence and Training
LTL-1005	5	126	Analytical Balances
LTL-1006	3	126	Refrigerator, Freezer, and Oven Thermometer Calibration and Maintenance
LTL-1007	3	126	Use of Instrument Records and Logbooks
LTL-1008	6	126	QC Corrective Action
LTL-1009	3	126	Blind Spike Program
LTL-1011	4	126	Procedures for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits
LTL-1012	2	126	Solvent QC Monitoring for Trace Residue Analysis
LTL-1013	4	126	Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials
LTL-1017	1	126	Internal Audit Procedures
LTL-1018	1	126	Overview of Review and Approval Practices for Validatable Data Packages
LTL-1019	2	166	Controlling, Maintaining, and Monitoring Laboratory Logbooks
LTL-1020	1	126	Integration of IC, GC, HPLC and GCMS Peaks
LTL-2001	6	166	Waste Segregation and Disposal
LTL-3202	3	166	Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081A/8082 by 3510C)
LTL-3302	2	126	Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Soil (8081A/8082 by 3550B)
LTL-4002	6	126	Electronic Sample Entry and Log-In
LTL-4103	0	126	Reviewing a Sample Delivery Group (SDG) and Updating Projects in the LIMS
LTL-4201	0	126	Package Deliverables for all Reporting Levels
LTL-7003	2	126	Inorganic Glass Cleaning Procedure

LABORATORY STANDARD OPERATING PROCEDURES (SOPs)
TABLE OF CONTENTS

SOP NUMBER	REV #	QAPP	TITLE
LTL-7015	1	126	Acid Digestion of Sediments, Sludges, and Soils Using SW846 Method 3050B
LTL-7202	4	126	Metals Analysis Using Inductively Coupled Plasma – Mass Spectrometry (ICP/MS) SW846 Method 6020
LTL-7501	2	166	Operation of the PE FIMS 400 for the Analysis of Hg.
LTL-8000	2	126	Determination of Retention Time Windows
LTL-8084	4	166	Analysis of Organochlorine Pesticides and PCBs by SW 846 Methods SW 8081A and 8082
LTl-8265	3	166	Determination of Volatile Organic Compounds by Method 8260B

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1001

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

Revision history:

Number	Date
7	12/2/05
6	8/25/04
5	6/25/02
4	5/3/99
3	7/22/94
2	9/16/87
1	7/31/86

Written by: Harry Romberg Date: 12/2/05
Harry Romberg, QA Officer

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

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 PURPOSE 3
1.2 SCOPE 3

2. PROCEDURES 3

2.1 WORD PROCESSING FORMAT 3
2.2 INITIATING AN SOP OR METHOD 3
2.3 REVISING AN SOP 5

3. ELEMENTS OF AN SOP/METHOD 5

3.1 ELEMENTS 5
3.2 SAVING THE DOCUMENT 9
3.3 AFTER COMPLETION OF THE DRAFT SOP 9

4. REFERENCES 10

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

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 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.

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 should 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,

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 (5.10.1.2, the 2003 NELAP standards have not been adopted by our accrediting authority at the time of this writing but include the same elements in section 5.5.4.1.2). 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.

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.

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.

3.1.18 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.

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.

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

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

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE **3**

1.1 PURPOSE 3
1.2 DOCUMENT TYPES 3
1.3 SCOPE 3

2. OPERATION PROCEDURES **3**

2.1 INITIATION, REVIEW AND UPDATING OF DOCUMENTS 3
2.2 TRACKING AND CONTROL OF EXISTING DOCUMENTS 5
2.3 STORAGE AND FILING OF CONTROLLED DOCUMENTS 10

APPENDIX A **12**

DOCUMENT CONTROL AND UPDATE FORM 12

APPENDIX B **14**

SOP DATABASE SCREENS 14

APPENDIX C **17**

SOP MANUAL DISTRIBUTION LOCATIONS 17

APPENDIX D **19**

CONTROLLED DOCUMENT STAMP 19
UNCONTROLLED STAMP 19
REVIEWED STAMP 19

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.

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

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.

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."

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.

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

"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.

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

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.

Appendix A

Document Control And Update Form

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: _____

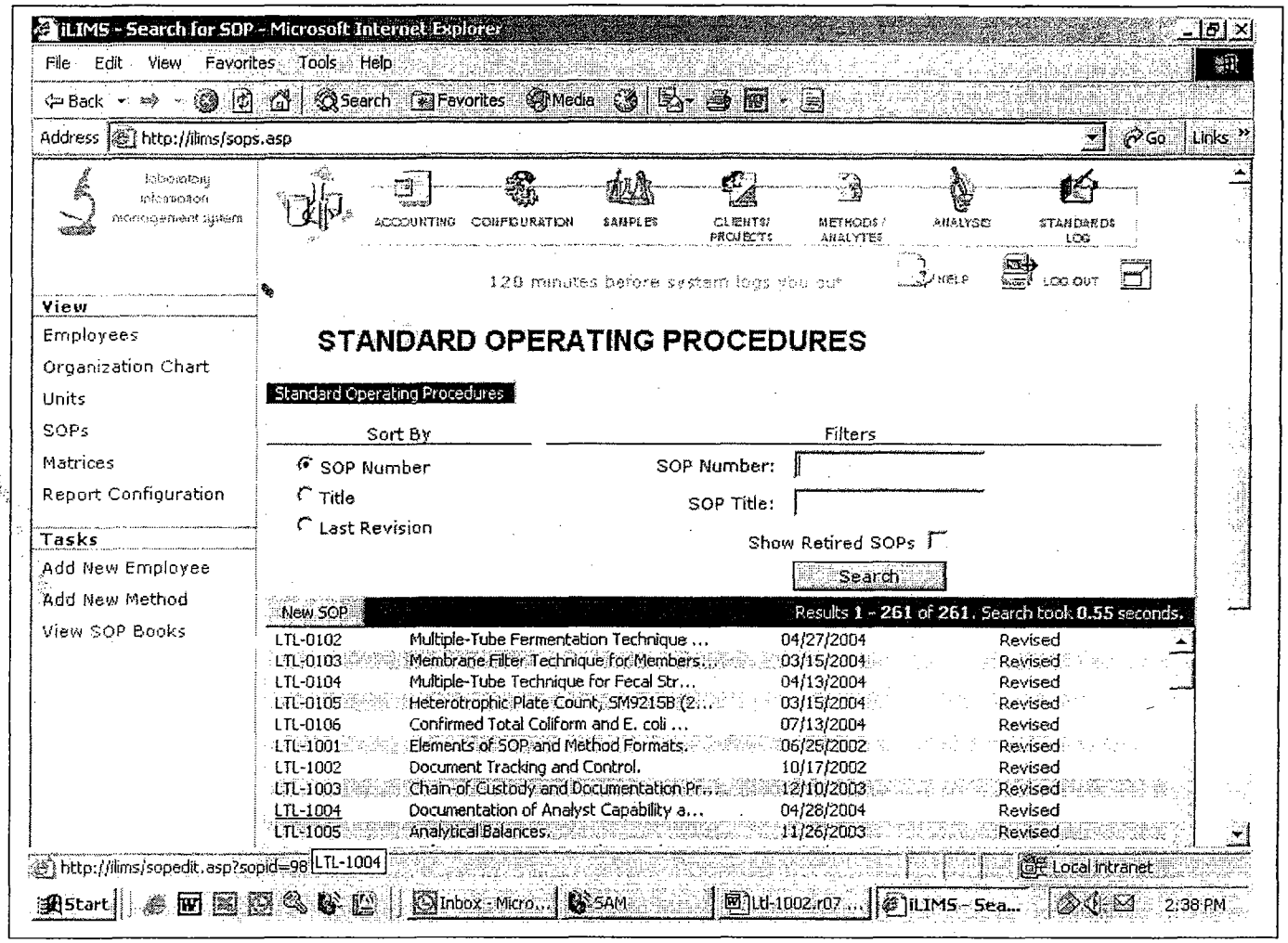
QA Approval: _____ Date: _____

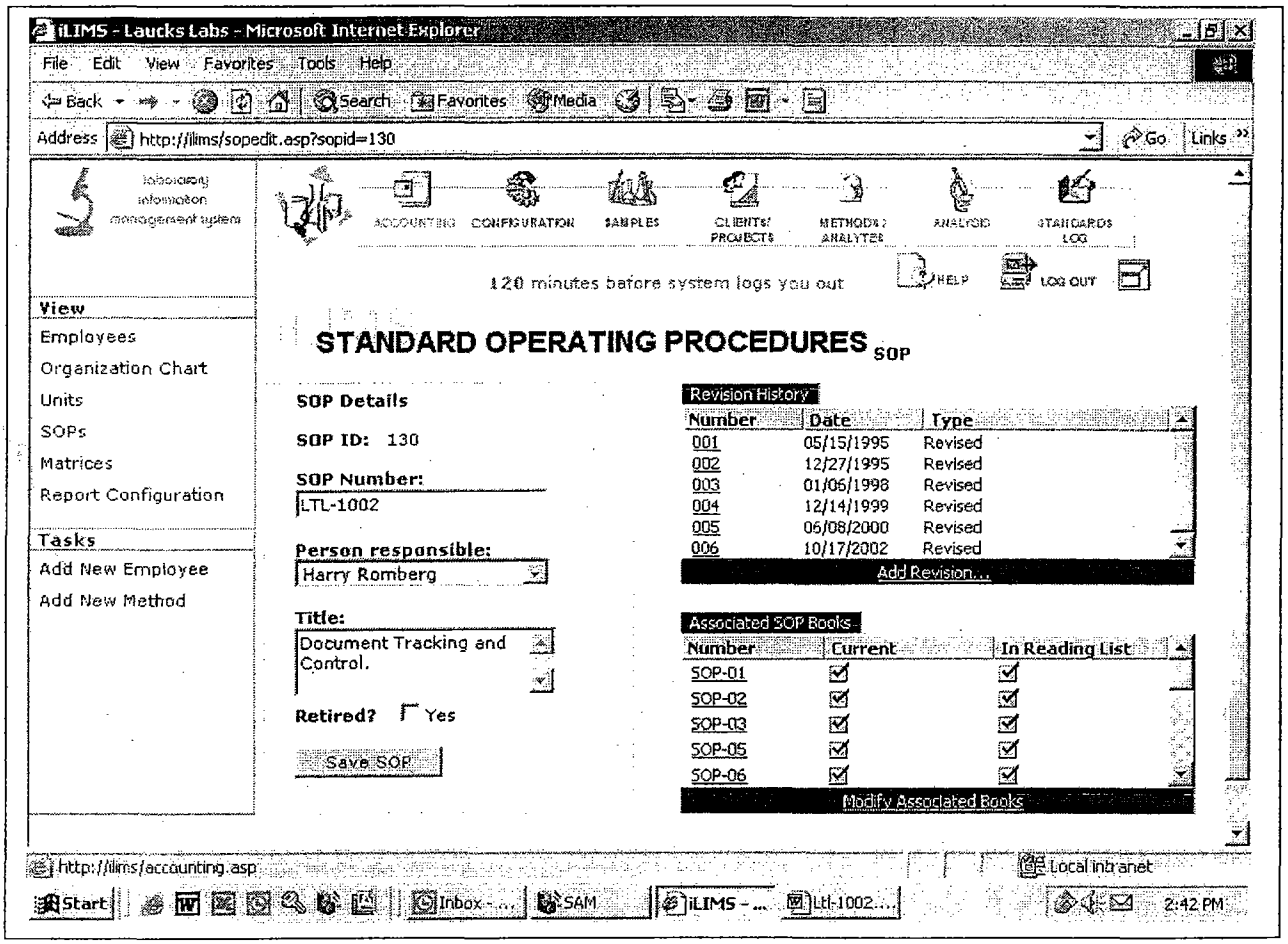
Implementation Date: _____

_____

Appendix B

SOP Database Screens

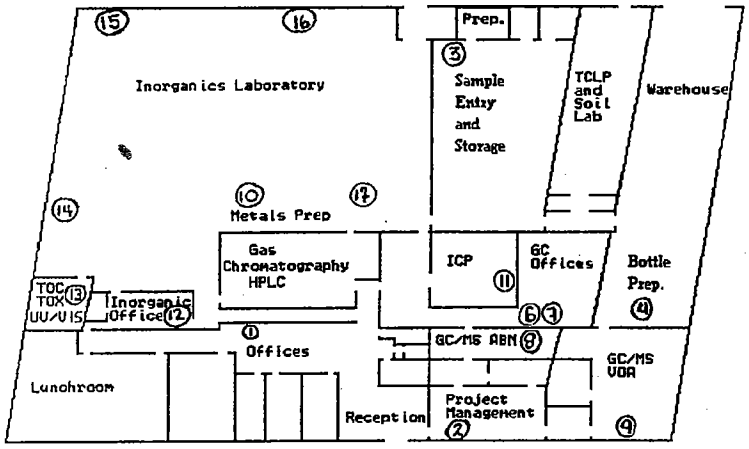

iLIMS - Search for SOP - Microsoft Internet Explorer
File Edit View Favorites Tools Help
Back Search Favorites Media
Address http://ilims/sops.asp
Go Links
ACCOUNTING CONFIGURATION SAMPLES CLIENTS/ PROJECTS METHODS / ANALYTES ANALYSES STANDARDS LOG
120 minutes before system logs you out
HELP LOG OUT
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
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
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

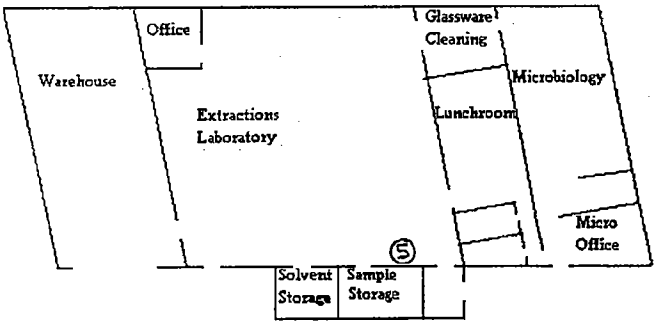
Appendix C

SOP Manual Distribution Locations

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



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



SOP Book Location

Appendix D

Controlled Document Stamp

Controlled Document

No. _____ Assigned to: _____

Uncontrolled Stamp

UNCONTROLLED

Reviewed Stamp

Reviewed

Does NOT require revision: ☐

Initials: _____ Date: _____

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

Written by: Harry Romberg Date: 6/7/05
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 6/8/05
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 Assigned to: TETRA

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 DESCRIPTION 3
1.2 DEFINITION OF TERMS 4

2. EQUIPMENT LIST 4

3. SAFETY PRECAUTIONS 4

3.1 SAFETY PRECAUTIONS 4

4. OPERATION PROCEDURES 4

4.1 IDENTIFICATION OF SAMPLES REQUIRING STRICT CHAIN-OF-CUSTODY 4
4.2 INITIATING INTERNAL CHAIN-OF-CUSTODY 5
4.3 MAINTAINING INTERNAL CHAIN-OF-CUSTODY 6
4.4 SAMPLE DISPOSAL AND CLOSING OF THE INTERNAL CHAIN-OF-CUSTODY 7
4.5 REVIEW OF CUSTODY LOGS 7

APPENDIX A 9

SECURE STORAGE CUSTODY LOG 9

APPENDIX B 11

VOLATILES CUSTODY LOGS 11

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 sample disposal. It does not address actual sample receipt, entry and log-in, nor does it address any aspect of samples analysis or reporting of results except as it pertains to maintaining the chain-of-custody. The chain-of-custody process is described only for samples requiring secure storage and strict chain-of-custody documentation.

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 the secure 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 forms provided 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 is transitioning into a more fully automated **L**aboratory **I**nformation **M**anagement **S**ystem we are calling iLIMS. This system includes bar-coding of samples, storage areas and even individuals' IDs thus enabling electronic tracking once samples have entered the lab. The physical handling of samples should remain the same except that instead of handwritten records, electronic versions will be used. At the present time, this system has not been fully implemented so greater detail will be provided in future revisions of this SOP, once the system has seen enough regular use to have stabilized and developed consistent practices.

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:

- 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 is maintained and documented throughout the period that the sample is in the possession of the laboratory. Any changes in the possession (custody) of the sample must be documented in order that the chain-of-custody can be properly maintained.

2. Equipment List

- Secure Storage Custody Log(s), see Appendix A
- Volatiles Custody Log(s), see Appendix B

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 Actual internal chain-of-custody procedures 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 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:

- 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.
- Initially, volatiles samples are stored in the refrigerator next to the 940 walk-in until they can be logged in and transferred to the outlying cold storage areas.
- The GC volatiles refrigerator is located in the area between the GC room and the laboratory computer system hub.
- The GC/MS volatiles refrigerator is located in the 940 warehouse area.
- The locked "cage" in the log-in area is for samples 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.

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 requiring secure storage are logged into any of these areas by the sample receiving representative using a Secure Storage Custody Log (Appendix A). Samples not requiring secure storage need not have this form completed. A custody log will be completed for each **workorder or SDG** for which samples require chain-of-custody procedures.

4.3 Maintaining Internal Chain-Of-Custody

4.3.1 When samples are logged out of storage areas, they will be signed out in the appropriate spaces by the person removing them.

- If they are being removed for analysis, the "**Action**" column should state the analyses being performed. When they are returned, the logsheet must also indicate such. Additionally, the "**Sample and Bottle Numbers**" column should indicate which samples are being removed for analysis (i.e. 1-10 metals digestion, or 3-5 NO3/NO2 analysis).
- Bottle numbers should also be recorded in the "**Sample and Bottle Numbers**" column.
- If they are being removed for transferal to another location (i.e. extractions), the "**Action**" column should state where they are being transferred. Additionally, the "**Sample and Bottle Numbers**" column should indicate which samples/bottles are being transferred (i.e. 1-10 volatiles, or 3-5 extractables).
- When samples are removed for final disposal, if all samples are being removed, the logsheet is signed and dated at the bottom of the page. If only certain samples are being disposed or to be even more clear, the "**Action**" column should indicate "disposed" and the "**Sample and Bottle Numbers**" column should indicate which samples/bottles are being disposed.

4.3.2 When samples are signed into another storage location, this is done using an identical Secure Storage Custody Log. Samples which are subsequently removed from these areas for analysis or disposal should be signed out using the same procedures as above.

4.3.3 Volatiles samples, being generally for a single analysis, are signed out using an abbreviated logsheet. Copies of GC/MS and GC VOA logsheets are located in Appendix B.

4.3.4 Any analyst removing samples from **any** secure storage area for the purpose of preparation or analysis or transferal to another department **must** sign the samples out using the Secure Storage Custody Log and must sign the samples back in when they are returned, or must sign them into another secure storage area. Samples must be in the possession of the analyst who signed them out at all times during this period and must not be left unattended. If samples are analyzed and then immediately disposed, as may be the case for some volatiles analyses, the "**Action**" column on the custody log should indicate "analysis and disposal." Note, in checking volatiles samples out using a volatiles custody log, it is assumed that the purpose is to analyze for volatiles since no other analysis is performed on these samples.

4.3.5 There is ample room on any one Secure Storage Custody Log in almost all cases to accommodate checking all sample containers for a particular SDG or workorder in and out as often as required for all of the pertinent analyses from that secure area. Should an additional page be required, it should be obtained from Sample Entry. A second (or third) page may not be initiated until all of the space on the previous form has been filled. The first page must be marked "1 of 2" in the upper right corner and the second page marked "2 of 2". In the unlikely event that even more pages are required, this mark can be crossed out (single line, initialed and dated) and the sheets marked "1, 2, or 3 of 3, etc.

4.4 Sample Disposal and Closing of the Internal Chain-Of-Custody

4.4.1 When samples have been signed out for final disposal the chain-of-custody process is considered to be complete. The Secure Storage Custody Logs must be collated, bound and turned in to the Quality Assurance Department in order that the chain-of-custody can be tracked for all samples requiring this process, should such tracking be required at a later date.

4.5 Review of Custody Logs

4.5.1 On at least a quarterly basis, supervisory personnel must review selected custody logs to insure they are being filled out properly and completely. The supervisor need not review all of the forms but must review at least 5% or 10 forms, whichever is greater. The supervisor will stamp the reviewed forms with a "reviewed by" stamp , initialing and dating the notation or may write "reviewed by" by hand, likewise initialing and dating the form. The supervisor should also cursorily review the remaining forms to make sure there are no blatantly obvious omissions but need not mark these forms unless a discrepancy is observed.

4.5.2 Review should include noting that all spaces are filled in properly and completely (this especially means that all samples that were checked out must be checked back in and that the person handling the samples must be identified), that errors are crossed out with a single line, initialed and dated, that entries are clear and not obliterated.

4.5.3 Should the supervisor find errors or omissions, they must issue a corrective action form to the individual who made the error (if it is obvious who that is) or to the appropriate supervisor. Any of these individuals can correct the form (initialing and dating the correction). The primary purpose of the corrective action notice is not intended to be punitive but is primarily so that the individual in error be re-trained as to the proper custody procedure.

4.5.4 Supervisors responsible for this review may assign the responsibility to other capable individuals but the ultimate responsibility is theirs. These supervisors are the Organics Division Manager for the volatiles logsheets, the Extractions supervisor for the

extractables, and the Sample Control supervisor for the main lab walk-in and unrefrigerated secure storage areas.

Appendix A

Secure Storage Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:_____ **LTL Number:**_____

Number of Containers (*optional*)**:**_____

Storage Unit:_____ **SDG Number** (*optional*)**:**_____

Sample and Bottle Numbers	Matrix (*optional*)	Location (*shelf*)	Logged Out			Logged In			Action
			Date	Time	By	Date	Time	By	

Samples Disposed of by _____ on _____

Appendix B

Volatiles Custody Logs

LAUCKS TESTING LABORATORIES
GC/MS VOA CUSTODY LOG

LABORATORY NUMBER	SAMPLE NUMBER	DATE & TIME REMOVED	BY	DATE & TIME RETURNED	BY	COMMENTS

LAUCKS TESTING LABORATORIES
GC VOA CUSTODY LOG

LABORATORY SAMPLES	ANALYSIS	DATE & TIME REMOVED	BY	DATE & TIME RETURNED	BY	COMMENTS

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1004

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

Revision history:

Number	Date
1.0	01/31/95
2.0	12/27/95
3	6/23/96
4	8/4/99
5	04/28/04
6	11/29/05

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

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

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 DESCRIPTION 3
1.2 SCOPE 3

2. DEFINITIONS 4

3. RESPONSIBILITIES 4

3.1 ANALYST 4
3.2 SUPERVISOR 5
3.3 QA 6
3.4 TRAINER 6

4. OPERATION PROCEDURES 6

4.1 OVERVIEW 6
4.2 TRAINING CHECKLISTS 7
4.3 SOP READING LISTS 7
4.4 DEMONSTRATIONS OF CAPABILITY 8
4.5 INITIAL AND CONTINUING DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENTS 9

5. REFERENCES 10

APPENDIX A 11

EXAMPLE TRAINING CHECKLISTS 14
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 14

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.

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.

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

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.

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

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.

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 <u>with no assistance</u>. 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.

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

Appendix A

Initial and Continuing Demonstration of Capability Certification Statements

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

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

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

Laucks 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.

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.

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.

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.

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.

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.

Laucks Testing Labs
Lachat Analyst Training Verification Checklist

Analyst Name:		Date:	Trainer:	Supervisor:	Analyst:
Documentation					
	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				
	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.

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.

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.

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.

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.

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.

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.

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.

Laucks 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.

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.

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

Revised by: Harry Romberg, QA Officer Date: 11-26-03

Approved by: Kathy Kreps, Laboratory Director Date: 12/29/03

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE **3**
1.1 GENERAL 3
2. EQUIPMENT LIST **3**
3. SAFETY PRECAUTIONS **3**
3.1 SAFETY 3
4. OPERATION PROCEDURE **4**
4.1 BALANCE SETUP 4
4.2 BALANCE AND WEIGHT CALIBRATION 4
4.3 RESPONSIBILITIES 5
4.4 DAILY CALIBRATION CHECK 6
4.5 WEEKLY CALIBRATION CHECK 8
4.6 ANNUAL CALIBRATION CHECK 9
5. REFERENCES **9**
APPENDIX I **10**
SAMPLE PAGE FROM A BALANCE LOGBOOK 10

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 a standard weight check 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**.

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.

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 the daily standard (a class "S" weight typical of the weight used on that balance) and

records the weight in the balance record book. If the 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. 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.

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 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.
- 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 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 weight of a size commonly used 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. He or she will also initial and date the entry (See Appendix I). The standard weight will be a class "S" weight or equivalent.

4.4.7 The daily weight, 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 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 must read within the limits of 0.9995 gm to 1.0005 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 30.0000 gm. Class "S" weight is then placed on the balance with the flask and the weight recorded. This second weight must read within the limits of 29.98 gm to 30.02 gm.

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 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.

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.

5. References

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

APPENDIX I

Sample Page from a Balance Logbook

	TV	Class "S" Read		Mon.	Tues.	Wed.	Thurs.	Fri	Sa
May 4 - May 9, 1998	2.0	2.00	Tare →	54.17	54.16	54.17	54.18	54.15	
	10.0	10.00	2.0 g. →	2.00	2.00	2.00	2.00	2.00	
DR	50.0	50.00		DR	DR	DR	DR	DR	
5-4-98	150.0	150.00		5-4-98	5-5-98	5-6-98	5-7-98	5-8-98	
May 11 - May 16, 1998	2.0	2.00	Tare →	54.17					
	10.0	10.00	2.0 g →	2.00					
DR	50.0	50.00		DR					
5-11-98	150.0	150.00		5-11-98					

Done once per week

Initial & Date required

Row Heading

Daily Weights

Initial & Date required

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

Written by: Harry Romberg, QA Officer Date: 6/13/05

Approved by: Kathy Kreps, Laboratory Director Date: 6/14/05

Controlled Document

No. 20 Assigned to: TETRA

Table of Contents

1. INTRODUCTION AND SCOPE **3**
1.1 METHOD DESCRIPTION 3
2. EQUIPMENT LIST **3**
2.1 EQUIPMENT 3
3. SAFETY PRECAUTIONS AND WASTE DISPOSAL **3**
3.1 SAFETY PRECAUTIONS 3
3.2 WASTE DISPOSAL 3
4. THERMOMETER PURCHASING AND IDENTIFICATION **4**
4.1 PURCHASING 4
4.2 IDENTIFICATION 4
5. CALIBRATION **4**
5.1 RECALIBRATION OF THE STANDARD THERMOMETER. 4
5.2 RECALIBRATION OF COLD STORAGE AND ROOM TEMPERATURE THERMOMETERS 5
5.3 RECALIBRATION OF OVEN AND OTHER THERMOMETERS 6
5.4 RECALIBRATION OF THE INFRARED THERMOMETER 7
5.5 DAILY CALIBRATION CHECK OF THE INFRARED THERMOMETER 8
6. MONITORING RESPONSIBILITIES **8**
6.1 USE OF CALIBRATION LOGS FOR COLD STORAGE MONITORING 8
6.2 MONITORING OVENS AND OTHER DEVICES 9
7. SPECIFICATION LIMITS AND CORRECTIVE ACTIONS **9**
7.1 THERMOMETER CRITERIA 9
7.2 THERMOMETER CORRECTIVE ACTIONS 10
7.3 COLD STORAGE CRITERIA 10
7.4 COLD STORAGE CORRECTIVE ACTIONS 10
APPENDIX I **12**
EXAMPLE CALIBRATION SPREADSHEET 12
APPENDIX II **15**
EXAMPLE COLD STORAGE TEMPERATURE LOG 15
APPENDIX III **17**
EXAMPLE INFRARED THERMOMETER DAILY CHECK LOG 17

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

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.

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.

4.2 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.

5.1.2 Note: 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 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.

5.2.3 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 be 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.

5.2.4 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.

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

5.2.6 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.

5.2.7 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.

5.2.8 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.

5.2.9 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.

5.2.10 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.

5.3 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 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 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.

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.

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 The calibration 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.

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.

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.

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

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).

Appendix I

Example Calibration Spreadsheet

Laucks Testing Laboratories
Thermometer Calibrations
2004/05

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
1	**Cold Storage ID**		**Location**		**Serial No.**		**Cal. Date**		**FC-53631 NIST reading**	**Thermom. Reading**	**Correction Factor**			
2	C01		Inorganics		ERTCO 4156		7/28/2004		3.9	5.2	-1.3			
3	C03		extractions		B B01679		7/28/2004		3.9	3.9	0.0			
4	C04		GC VOA		B B03987		7/28/2004		3.9	4.1	-0.2			
5	C05		Inorganics		B02580		7/28/2004		3.9	3.8	0.1			
6	C06		Warehouse		B B04765		7/28/2004		3.9	3.8	0.1			
7	C07		940 warehouse		VWR 172101		7/28/2004		3.9	2.8	1.1			
8														
9	R01		Sample Entry		A08946		7/28/2004		3.9	3.8	0.1	broken 11/2/2004		
10	R02		GC Semi Stds.		B B03534		7/28/2004		3.9	3.8	0.1			
11	R04		GC/MS whse.		B B00906		7/28/2004		3.9	3.9	0.0			
12	R06		extractions		B B01708		7/28/2004		3.9	3.9	0.0			
13	R07		extractions		B B04059		7/28/2004		3.9	4.2	-0.3			
14	R08		Inorganics		B 03021		7/28/2004		3.9	3.7	0.2			
15	R11		929 warehouse		B B01342		7/28/2004		3.9	3.8	0.1	moved to R01 on 11/2/2004		
16														
17	W01		940 Walk-in		B B03928		7/28/2004		3.9	4.2	-0.3			
18	W02		921 Walk-in		B B01919		7/28/2004		3.9	3.5	0.4			
19														
20	F01		GC/VOA		B B B08543		7/27/2004		-12.2	-12.2	0.0			
21	F03		GC semivolatiles		B B B08959		7/27/2004		-12.2	-12.2	0.0			
22	F05		GC/MS VOA		Ertco 1152		7/27/2004		-12.2	-12.0	-0.2			
23	F06		GC semivolatiles		Ertco 5169		7/27/2004		-12.2	-12.5	0.3			
24	F07		GC/VOA stds.		B B B08765		7/27/2004		-12.2	-11.5	-0.7			
25	F09		GC Instrument Room		Ertco 5034		7/27/2004		-12.2	-12.0	-0.2			
26			spare		Ertco 5236		7/27/2004		-12.2	-11.5	-0.7			
27			spare		Ertco 1838		7/27/2004		-12.2	-11.0	-1.2			
28			spare		VWR H21301		7/27/2004		-12.2	-11.0	-1.2			
29			spare		Ertco 1535		7/27/2004		-12.2	-10.5	-1.7			
30			spare		B B B09215		7/27/2004		-12.2	-14.5	2.3			
31			spare		Ertco 2034		7/27/2004		-12.2	-10.5	-1.7			
32														
33	Hg thermometer		Sample entry		BCR 2		7/28/2004		3.9	3.8	0.1			
34	IR Thermometer (ITT-330)				Horiba 226099		7/29/2004	**Glass**			**(at E=86)**			
35							7/29/2004	**Plastic**			**(at E=89)**			
36	IR Thermometer (ITT-330)				Horiba 132013		7/29/2004	**Glass**			**(at E=85)**			
37							7/29/2004	**Plastic**			**(at E=90)**			
38					ERTCO 5124		7/28/2004		3.9	4.2	-0.3			
39					ERTCO H93-203		7/28/2004		3.9	4.5	-0.6			
40					ERTCO 2920		7/28/2004		3.9	4.0	-0.1			
41					ERTCO H93-102		7/28/2004		3.9	5.0	-1.1			
42					unmarked Temp Chex		7/28/2004		3.9	4.1	-0.2			
43	**Oven/Water-bath/Other ID**		**Location**		**Serial No.**		Cal. Date		**FC-53631 NIST reading**	**Thermom. Reading**	**Correction Factor**			

Laucks Testing Laboratories
Thermometer Calibrations
2004/05

	A	B	C	D	E	F	G	H	I	J	K	L	M	N
44	VWR 1320		Extractions		8						0			
45	VWR 1310				3						0			
46	VWR 1330F		#4 Univ. Enterprises		L12-004						0			
47	VWR 1370F				F42653						0			
48	Thelco				VWR 02429						32	Deg F		
49	VWR 1300 U (TOC room)				VWR 61066-046						0			
50	Pensky Martin (AST		VWR 61091-001		IY7422						32	Deg F		
51	VWR 1305U				National Appliance #7						0			
52	VWR 1300 U (Extractions)				F22592						0			
53	Blue M (Extractions)				VWR 61066-104						0		Broken	
54	Blue M (Extractions)				F22716						0			
55	front waterbath				Enviro-Safe 6									
56	Napco Oven				SPER Sci.						0			
57	Digestion Area		VWR 61014-949		9						0			
58	VWR ASTM 9F				IY2085						32	Deg F		
59	ERDCO		Setaflash		1SF5531/85A038						32	Deg F		
60	ERDCO		Setaflash		1SFA5531/151831						32	Deg F		
61	**Oven/Water-bath/Other ID**		**Location**		**Serial No.**		**Cal. Date**		**FC-53631 NIST reading**	**Thermom. Reading**	**Correction Factor**			
62	metals digestion				00-01			broken ?????			0.0			
63	BOD thermometer		BOD Incubator		BCR 1						0.0			
64														
65	spare				F22731						0			
66														
67	VWR ASTM 9F				09107						32.0	Deg F		
68														
69			VWR 61222-504		20						0			
70			VWR 61222-504		21		disposed 10/22/04				0			
71	Hg mod block										0			
72			VWR 61222-504		23						0			
73			VWR 61222-504		25						0			
74			VWR 61222-504		26						0			
75			VWR 61222-504		27						0			
76			VWR 61222-504		28						0			
77			VWR 61222-504		29						0			
78			VWR 61222-504		30						0			
79			VWR 61222-504		31		11/8/2004		99.5	102.0	-3			
80			VWR 61222-504		32		11/8/2004		99.0	101.0	-2			
81			VWR 61222-504		33		11/8/2004		99.0	101.0	-2			
82			VWR 61222-504		34		11/8/2004		100.0	104.5	-5			
83			VWR 61222-504		35		11/8/2004		100.0	102.0	-2			
84														
85			Extractions oven		T6920						0			

Appendix II

Example Cold Storage Temperature Log

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **2005** 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.

Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/29/04

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**

Any and all a[illegible] **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of th[illegible] with the date the action occurred.

Samples **MUS[illegible]T** be stored in units which are not maintaining the proper temperature.

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: **2005** 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										
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\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 t[illegible]meter is not working properly, the glass thermometer **must** be used.

Any and all a[illegible] **MUST** be recorded on this log sheet. If there is insufficient room, mark on the back of th[illegible] with the date the action occurred.

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

Revised by: Harry Romberg, QA Officer Date: 9/23/05

Approved by: Kathy Kreps, Laboratory Director Date: 9/23/05

Controlled Document

No. 20 **Assigned to:** TETRA

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 INTRODUCTION 3
1.2 SCOPE 3
1.3 DEFINITION OF TERMS 3

2. EQUIPMENT LIST AND STANDARDS 3

2.1 EQUIPMENT 3

3. OPERATION PROCEDURES 3

3.1 ALL LOGBOOKS 3
3.2 MAINTENANCE MANUALS 4
3.3 INSTRUMENT RUN-LOGS 5
3.4 OTHER LOGBOOKS 7

4. REFERENCES 7

APPENDIX I 8

GC/MS RUN LOGS 8

APPENDIX II 12

GC/VOA & HPLC RUN LOGS 12

APPENDIX III 15

GPC RUN LOG 15

APPENDIX IV 17

ION CHROMATOGRAPHY RUN LOG 17

Controlled Document
No. _____ Assigned to: _____

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 log, 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

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 logbooks must be altered by a single-line crossout which must also be initialed and dated. No erasures, overwriting, white-out or multiple-line crossouts (blacking out) are acceptable.

3.1.3 **NOTE:** Empty space in 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.

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 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 from each laboratory workorder 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.

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. 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 <u>in the order that they were analyzed</u>.

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 often in use in the Inorganics area of the laboratory.

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.

3.3.10 Information critical to identifying the analytical run (date, analyst, analysis type) must be included in the header information. If multiple analytical runs were made in one day, they must be identified as run #1, run #2, etc. or by some clear identifying mechanism to

indicate which run was which. 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.

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*

Appendix I

GC/MS Run Logs

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 IRW 5/25/04

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 5G/5ML+IS/SS(MV8-16-14)	24 May 2004 09:33
F0524003	1.00	1.00	1.00	BFB50NG 2UL(MV8-11-3) DIRECT INJ	24 May 2004 10:11
F0524004	1.00	1.00	1.00	BFB50NG 2UL(MV8-11-3) DIRECT INJ	24 May 2004 10:31
F0524005	1.00	1.00	1.00	BFB50NG 2UL(MV8-11-3) DIRECT INJ	24 May 2004 10:50
F0524006	1.00	1.00	1.00	VSTD010 25ML+std(MV8-17-11)+IS/SS	24 May 2004 11:21
F0524007	1.00	1.00	1.00	S052404MWOF1 25ML+IS/SS+MS(MV8-15-20)	24 May 2004 12:00
F0524008	1.00	1.00	1.00	B052404MWOF1 25MLpfw+IS/SS(MV8-16-14)	24 May 2004 12:34
F0524009	1.00	1.00	1.00	B052404MWOF2 25MLpfw+IS/SS(MV8-16-14)	24 May 2004 13:08
F0524010	1.00	1.00	1.00	S052404MWOF2 25ML+IS/SS+MS(MV8-15-20)	24 May 2004 13:42
F0524011	1.00	1.00	1.00	0405247-01 KCSW 25ML+IS/SS	24 May 2004 14:41
F0524012	1.00	1.00	1.00	0405247-02 KCSW 25ML+IS/SS	24 May 2004 15:15
F0524013	1.00	1.00	1.00	0405247-03 KCSW 25ML+IS/SS	24 May 2004 15:49
F0524014	1.00	1.00	1.00	0405246-01 KCSW 25ML+IS/SS	24 May 2004 16:23
F0524015	1.00	1.00	1.00	0405246-02 KCSW 25ML+IS/SS	24 May 2004 16:57
F0524016	1.00	1.00	1.00	0405246-03 KCSW 25ML+IS/SS	24 May 2004 17:31
F0524017	1.00	1.00	1.00	0405268-01DL KCSW 1:100 25ML+IS/SS	24 May 2004 18:06
F0524018	1.00	1.00	1.00	0405268-02 KCSW 25ML+IS/SS	24 May 2004 18:40
F0524019	1.00	1.00	1.00	0405271-01 KCSW 25ML+IS/SS	24 May 2004 19:14
F0524020	1.00	1.00	1.00	0405271-02 KCSW 25ML+IS/SS	24 May 2004 19:48
F0524021	1.00	1.00	1.00	0405289-01 KCSW 25ML+IS/SS	24 May 2004 20:22
F0524022	1.00	1.00	1.00	0405289-02 KCSW 25ML+IS/SS	24 May 2004 20:57
F0524023	1.00	1.00	1.00	0405290-01 KCSW 25ML+IS/SS	24 May 2004 21:31

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

Appendix II

GC/VOA & HPLC Run Logs

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 #

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 14:2
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 22:5
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

Appendix III

GPC Run Log

Laucks Testing Labs, Inc.
GPC Bench Sheet

Date	5-5-04
Cal. Ref	050304GPC2
Analyst	AGH
Case #	
SDG #	ATPØ1
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						
17						
18						
19						
20						
21						
22						
23						

5-5-04

AGH

Comments: $MeCl_2$ (B&S #C5542), Filter lot # 5252

Appendix IV

Ion Chromatography Run Log

Method:IC042804
Matrix:Water
Analysis Date:04/29/04

Laucks Testing Laboratories
Method 300.0,9056
Analyst:Kelly Bottem

CCV: 250uL of Cal Std IC7*7*7
MS/MSD: 100 uL of Cal Std IC7*7*7
ICV/SRM: 25 uL of 7 Anion Standard IC7*4*17

Sample Name	Dilution	Fluoride	Chloride	Nitrite	Nitrate	Sulfate
icv/srm ic7*4*17	**20**	**20.7571**	**28.1188**	**31.15**	**22.069**	**141.454**
icb	**1**	**0**	**0**	**0**	**0**	**0**
0404503-01*1	1	0.305714	6.5338	0	0.9333	7.00319
0404503-01DL1	10	1.58431	7.1992	0	1.3716	10.4837
0404498-01*1	1	0.237328	4.00286	0	0	1.52455
0404498-01DL1	10	0	4.14528	0	0	0
0404375-02*1	1	0.20221	7.23151	0	1.1159	14.5864
0404375-02DL1	10	0	6.70956	0	1.2786	17.4677
0404505-01*1	1	0.206999	0.76232	0	0	24.3554
0404505-01DL1	10	0	90.3559	0	0	26.4908
0404505-02*3	1	0.163571	5.5079	0	0	93.6522
0404505-02DL1	10	0	5.52087	0	0	82.8151
ccv1 ic7*7*7	**1**	**1.9472**	**4.87496**	**0.9882**	**1.9379**	**9.81969**
ccb1	**1**	**0**	**0**	**0**	**0**	**0**
0404505-04*7	1	0.174273	26.261	0	0.196	27.7629
0404505-04DL1	10	0	22.2386	0	0	29.3402
0404505-05*9	1	0.163918	9.89398	0	0.4178	11.2105
0404505-05DL1	10	0	9.62529	0	0.8275	13.7471
0404505-06*11	1	0.166611	6.40856	0	0.5779	14.8136
0404505-06DL1	10	0	6.49043	0	1.0035	17.7503
0404505-07*13	1	0.170444	1.12308	0	0.5678	2.85433
0404505-07DL1	10	0	1.86015	0	1.026	0
0404505-08*15	1	0.173858	11.7743	0	0.2273	24.6864
0404505-08DL1	10	0	11.2517	0	0.7812	27.1834
ccv2 ic7*7*7	**1**	**1.93819**	**4.76516**	**0.9985**	**1.9088**	**9.58808**
ccb2	**1**	**0**	**0**	**0**	**0**	**0**
0404505-03*5	1	0.172442	4.61838	0	0.2405	19.4093
0404505-03DL1	10	0	4.50283	0	0	20.9725
0404505-09*17	1	0.174666	23.6637	0	0.4024	69.7781
0404505-09DL1	10	0	20.061	0	0.8294	51.553
0404505-10*19	1	0.32822	9.86459	0	0.1523	18.5545
0404505-10DL1	10	0	9.11546	0	0	21.0666
0404505-11*21	1	0.208646	13.7491	0	0.2295	27.6316
0404505-11DL1	10	0	18.5258	0	1.0663	29.5105
0404505-12*23	1	0.176576	24.2449	0	0.4479	72.5614
0404505-12DL1	10	0	20.0165	0	0.8149	63.1476
ccv3 ic7*7*7	**1**	**1.94091**	**4.68773**	**0.978**	**1.9042**	**9.59396**
ccb3	**1**	**0**	**0**	**0**	**0**	**0**
0404505-13*26	1	0	0	0	0	0
0404458-01*1	10	0	20.0298	0	0	23.5097
0404460-01*1	10	0	17.3769	0	0	19.618
0404459-01*1	1	0.250771	7.92812	0	0	9.61237
0404459-01DL1	5	0	3.69725	0	0	6.3902
0404459-01ms	**5**	**3.97751**	**12.5902**	**1.852**	**3.8602**	**23.6239**

KBW 5/5/04

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

Written by: Harry Romberg Date: 4/21/05
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 4/21/05
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 Assigned to: Jetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 PURPOSE 3
1.2 SCOPE 3

2. DEFINITION OF TERMS 3

2.1 THIS SECTION DEFINES TERMS AND ACRONYMS AS THEY ARE USED IN THIS SOP 3

3. OUT-OF-CONTROL EVENT PROCEDURE 4

3.1 IDENTIFYING AN OUT-OF-CONTROL EVENT 4
3.2 RESPONDING TO AN OUT-OF-CONTROL EVENT 5
3.3 CORRECTIVE ACTIONS 7
3.4 DOCUMENTING AN OUT-OF-CONTROL EVENT 8

APPENDIX 1 11

CORRECTIVE ACTION FORM 11

APPENDIX 2 13

QC_DB REPORT FORM 13

APPENDIX 3 15

OUT-OF-CONTROL EVENT FORM 15

APPENDIX 4 17

EXAMPLE SAMPLE RECEIPT FORMS 17

APPENDIX 5 20

EXAMPLE LOGSHEET (COLD STORAGE) 20

1. Introduction and Scope

1.1 Purpose

1.1.1 The purpose of this SOP is to establish a system to identify, document and resolve out-of-control events.

1.2 Scope

1.2.1 An out-of-control event may be recognized by any member of Laucks. When they occur, the analyst, supervisor and Quality Assurance work jointly to solve and correct the problem. Out-of-control events are documented using an Out-of-Control-Event form or a Corrective Action form, 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 this action is elucidated in an SOP specific to that process.

1.2.2 At the time of this writing, Laucks is currently in the process of changing Laboratory Information Management Systems (LIMS). This will likely lead to some changes in forms, nomenclature and practices which have not yet been developed. Though some of the specifics will change, this SOP still serves the purpose of defining the concepts involved. When the new system has been implemented and changes have stabilized enough, this SOP will be revised to reflect the changes that may result from the new system.

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.

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. Out-Of-Control Event Procedure

3.1 Identifying an Out-Of-Control Event

3.1.1 The following is a list of examples of out-of-control events. This is not a complete list of all possible out-of-control events 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).

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 corrective action 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 or 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

3.2 Responding to an Out-Of-Control Event

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.

3.2.2.3 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.

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 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 type of analysis, the extent of the discrepancy, and whether the event is determinant or not. 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.

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 and may not even require the completion of any related out-of-control event forms.

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.

3.4 Documenting an Out-Of-Control Event

3.4.1 This is accomplished by completing one of the following

- A Corrective Action (CA) Form (See Appendix 1)
- A QC_DB Report Form (for Inorganics analytical QC only, see Appendix 2)
- An Out-Of-Control Event (OOCE) Form (lab use only, see Appendix 3)
- A Sample Receipt Form (for sample receipt events, see Appendix 4)
- 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 5).

3.4.2 CA forms are general and are for documenting corrective action taken to correct problems not associated with a particular analytical event. 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.)

3.4.3 Out-Of-Control Event (OOCE) Forms are filled out by **technical** laboratory staff only and are designed for documenting analytical QC failures and associated corrective actions. Where other forms, such as the Inorganics QC_DB Report Form, are used to document that the QC parameters were checked, any failures of QC and the decision to perform corrective action or continue data processing must be documented on the OOCE form. The checklist may then be attached to the OOCE form for final data submission.

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.

Note: QC_DB report forms will most likely be forms which will be dropped with implementation of previously mentioned the new LIMS.

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 OOCE 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 OOCE form provides check boxes for most analytical events.

Note: Holding time violations due to laboratory error are annotated on the OOCE form. 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 OOCE Form 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 OOCE Form 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 OOCE forms must be turned into QA. Copies must be included in each SDG or workorder in validatable packages and in the first workorder in the "samples affected" column for non-validatable data packages.

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. OOCE forms must be turned in with the data and the supervisor creating the narrative comment for that area will comment on any decisions resulting from failed QC which could impact data validity or interpretation.

Appendix 1

Corrective Action Form

Laucks Testing Laboratories
Corrective Action Report

1) Problem Description:

Response tasked to: _____ on _____

By: _____ Response Requested By _____

2) Cause:

3) Action Taken:

Completed by _____ on _____

☐ Corrective actions will be reviewed 30 days after completion to verify problem has been corrected.

☐ No further action necessary

Reviewed by: _____ on _____

1) Person initiating corrective action fill out Part 1 and may fill out Part 2 if they are aware of the cause
2) Original goes to person tasked with a response; one copy goes to QA Officer and another kept by person initiating corrective action
3) Person tasked completes response in Part 2 (if not previously completed) and Part 3, signs response, and returns original to person initiating action
4) Person initiating action determines if action corrects the problem and signs "Reviewed by." If action was insufficient, return to the person charged with responding without signing.
5) Completed original goes to QA Officer

Appendix 2

QC_DB Report Form

Laucks Testing Laboratories

QC_DB Report Form

Analyst_____

Checker_____

Test Code_____

		QC Exceeds Control Limit √ if yes	Corrective Action Approved By ____
PBlk	B_____05_____	☐	
MS/MSD	K_____05_____	☐	
SRM	R_____05_____	☐	
Blk Spk	S_____05_____	☐	
MS/Dup	M_____05_____	☐	
Duplicate	D_____05_____	☐	
BS/BSD	L_____05_____	☐	

This report validates the following work orders

Appendix 3

Out-Of-Control Event Form

OUT OF CONTROL EVENT FORM

No: _____

Date Recognized: _____
Date Occurred: _____
Method: _____
Initiated By: _____
Analyst: _____

- ☐ GC/MS VOA
- ☐ GC/MS ABN
- ☐ GC VOA
- ☐ GC non-VOA
- ☐ HPLC
- ☐ Metals
- ☐ Wet Chemistry
- ☐ Extractions
- ☐ Data Management

Samples Affected (Workorder & Sample Numbers.)

Type of Event: (*check all that apply*)

___ Holding time missed (*describe below*)
___ Blank ≥ MDL___ RL___ CRQ/DL___
___ Spike Recoveries do not meet criteria
___ Duplicate RPDs do not meet criteria
___ MS/MSD Results do not meet criteria ___ %Rec ___ RPD
___ BS/BSD Results do not meet criteria ___ %Rec ___ RPD
___ Analytical Spike recoveries do not meet criteria
___ Standard Additions do not meet criteria
___ LCS or Blank Spike Recoveries do not meet criteria
___ Surrogate Recoveries do not meet criteria
___ Calibration Corr. Coefficient does not meet criteria
___ Calibration Verification does not meet criteria ___ Init ___ Cont.
___ Recovery ___ Retention time ___ %D
___ Tuning fails criteria
___ ISTD fails criteria
___ Calculation/Transcription error
___ Other (explain)

Corrective Action: (*check all that apply*)

___ Repeat Calibration
___ Made new standards
___ Reanalyzed, Date: _____
___ Sample(s) Redigested/Reextracted Date: _____
___ Results Recalculated
___ Cleaned System
___ Ran Standard Additions
___ Notified Client _____
___ Other (Please explain)

Check One:

___ Original Results Reported
___ Rerun Results Reported

Notified:

___ QA
___ Client Services

Action taken By: _____ Date: _____ Reviewed by Initiator: _____ Date: _____
___ Out of Control Event Corrected By: _____

___ Corrective Actions Not Successful (signatures required) **DATA MUST BE FLAGGED AND/OR NARRATED**

Analyst: _____ Date: _____
Supervisor: _____ Date: _____
QA Department: _____ Date: _____

Distribution:
Original to QA
Copy to workorder / SDG file for all validatable packages and to first workorder on list for non-validatable d

Appendix 4

Example Sample Receipt Forms

Laucks Testing Laboratories, Inc.
NON-CLP SAMPLE RECEIPT LOG

Initial once samples are checked in_____

DATE RECEIVED:_____ SAMPLE LOG-IN DATE:_____
TIME RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____ CLIENT PROJECT:_____
SDG #_____ AIRBILL ATTACHED?:(#)_____
COC #_____ RECEIVED BY:_____

Non-Conformance: (Check applicable item(s)) Client IDs affected:

- ☐ (1) Not enough sample sent for proper analysis. #s affected:_____
- ☐ (2) Sample Bottle received broken and/or cap not intact._____
- ☐ (3) Custody seal: Absent____ Present/Intact_____ Present/Broken _____
- ☐ (4) Any temperature out of compliance:_____
- ☐ (5) Sample received outside of holding time._____
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used._____
- ☐ (7) Illegible sample numbers or label missing from bottles._____
- ☐ (8) Identification on bottle same as identification on paperwork: yes:_____no:_____
- ☐ (9) Incomplete instructions received with sample(s), i.e., no Request for Analysis, no Chain-of-Custody._____
- ☐ (10) Samples received in improper container._____
- ☐ (11) Samples held in field before receipt by Lab. Days (specify)_____
- ☐ (12) Air Bubble(s) in ___of ____ samples for volatiles analysis._____
- ☐ (13) Other _____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____
☐	(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	_____	_____
☐	(7) Other (Specify)_____	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (2) In-house

P:\PM\Holly\docs\fax\pjmemo.doc12/22/94

DATE RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____
RECEIVED BY:_____

Non-Conformance:

☐ Other

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

_____

CORRECTIVE ACTION: (Check applicable item(s))

		Correction action taken by: Inititals	Date
☐	(1) Client informed verbally (Client Services).	_____	
☐	(2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐	(3) Sample processed "as received" (Sample Entry).	_____	_____
☐	(4) Re-sampling requested of client (Client Services).	_____	_____
☐	(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

_____

_____

_____

_____

_____

Appendix 5

Example Logsheet (Cold Storage)

Cold Storage Temperature Log

Laucks Testing Laboratories, Inc.

Cold Storage ID #:_____ Thermometer ID:_____

Location:_____ Year: **2005** 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.

Q:\TEMPLATS\LTLFORMS\REFERFRM.DOC 12/29/04

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.

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.

Control Limits

Refrigerators	**4°C ± 2°C**
Freezers	**-10°C to -20°C**
BOD Incubator	**20°C ± 1°C**

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1009

Title: **Blind Spike / PE Sample Evaluation Program**

Revision history:

Number	Date
LTL-0048 Rev1.1	5/18/92
2	6/21/96
3	6/19/00

Written by: Harry Romberg Date: 6-19-2000
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 06-19-00
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. Introduction and Scope 3
    1.1 Description 3
    1.2 Definition of Terms 3
2. Equipment List and Standards 3
    2.1 Equipment 3
    2.2 Reagents 3
3. Safety precautions and Waste Disposal 4
    3.1 Safety Precautions 4
    3.2 Waste Disposal 4
4. Materials 4
    4.1 Sources 4
    4.2 Storage 5
5. Operation procedures 5
    5.1 Requirements and Scheduling 5
6. Evaluation and Reporting 7
    6.1 Data Package Organization 7
    6.2 Evaluation 8
    6.3 Remedial Actions 8
7. Record Keeping 9
    7.1 Analyst and Method 9

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 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 with a value which is not known.

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 unknown amount of target analyte.

2. Equipment List and Standards

2.1 Equipment

2.1.1 Pipets, flasks, containers etc. necessary to prepare spikes for submission.

2.2 Reagents

2.2.1 Deionized water, methylene chloride and other solvents or preservatives that may be required to prepare spikes. Some samples may be prepared by outside sources and 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 health hazards. The health effects of these various chemicals may be ascertained by reading the appropriate material safety data sheets (MSDS). 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 waster disposal.

4. Materials

4.1 Sources

4.1.1 Materials may be WS, WP or other materials from an external performance evaluation. 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), SPEX, Restek, Supelco or any other reputable vendor.

4.1.3 Materials may be purchased either as Performance Evaluation 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 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 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 Performance Evaluation (PE) sample. 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 performance by analyzing a single blind PE sample.

5.1.4 PE 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 PE samples may be from a performance evaluation study, such as a Water Pollution (WP) or Water Supply (WS) study, an independent vendor PE, such as Environmental Resource Associates (ERA) or Analytical Products Group (APG), or it may be prepared by an area supervisor from a known material. Blind PE samples will almost always be prepared as aqueous solutions except in limited circumstances, such as fuel hydrocarbons, where soil samples are periodically analyzed. Other ERA, APG or independent sources of materials will be used where components are not present in WP, WS, DMRQA or other "official" PE samples. Acceptable results from programmatic samples, such as those for AIMTech, Army Corps of Engineers, or NFESC may be used to qualify analysts or to otherwise demonstrate performance, even though in some instances an actual value may not be provided by the agency.

5.1.6 WP and WS program samples are currently analyzed semiannually and DMRQA samples approximately annually. Supplementary PE samples for analytes not present in these samples (such as fuels or GC/MS semivolatiles) are generally obtained from the same source (APG, ERA or a similar vendor). Other external PE samples from programs such as NFESC, AIMTech, or the Army Corps of Engineers may be analyzed at the discretion of those programs but be used for evaluation. The precise schedule for submittal of all but programmatic samples is at the discretion of QA in order to meet laboratory needs to qualify analysts or methods or to meet other requirements. At the time of the latest revision of this SOP, both state and federal agencies are still determining the implication to their programs of WP, WS & DMRQA sample privatization. In addition, the laboratory is still evaluating the costs associated with purchasing these samples commercially. Once the requirements, costs and scheduling become more clear the result may be that there will be an overall reduction in the total number of PE samples the laboratory purchases in order to fulfill program requirements within a reasonable budget and the schedules may be modified at the discretion of QA.

5.1.7 One set of PE 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 PE sample contains one or more components from a multi-component analysis (such as a semivolatiles or pesticide mixture), the analytes themselves must not be divulged.

5.1.9 Blind spikes should be analyzed in at least duplicate, and frequently 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 performance evaluation program.

5.1.10 Blind spikes are typically determined for the following analyses (in water excepts as noted):

- ICP metals
- ICP/MS metals
- Graphite furnace metals (optional)
- Mercury

- GC Volatiles
- Gas/BTEX water & soil
- Diesel water & soil
- Petroleum Hydrocarbons (418.1) water & soil
- Pesticides
- GC/MS Volatiles
- GC/MS Semivolatiles
- PNAs
- Cyanide
- Total Organic Halogens
- Total Organic Carbon
- Phenolics
- Ion Chromatography (F, Cl, NO_3, SO_4)
- NO_3/NO_2 Automated Cd reduction
- others at the discretion of QA

5.1.11 Where other method references are very similar to those above, the same PE analysis may be considered adequate documentation for both methods. Other blind PE 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.

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 PE 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 90% of the target analytes are quantified correctly as long as all are at least identified.

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.

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 PE 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 performance.

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. Method evaluations will be kept separately but may mirror the analyst's evaluation.

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**

Revision history:

Number	Date
7	11/29/05
6	6/28/05
5	12/31/03
4	1/30/98
3	6/21/96
2	1/08/96
1.1	6/15/90

Revised by: Harry Romberg Date: 11/29/05
Harry Romberg, QA Officer

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

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 OVERVIEW 3
1.2 METHOD DESCRIPTION 5
1.3 DEFINITION OF TERMS 7

2. EQUIPMENT LIST AND STANDARDS 9

2.1 EQUIPMENT, REAGENTS AND STANDARDS 9

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 10

3.1 SAFETY PRECAUTIONS 10
3.2 WASTE DISPOSAL 10

4. CALIBRATION AND QUALITY CONTROL 10

5. RESPONSIBILITIES 10

5.1 ANALYST 10
5.2 DEPARTMENT MANAGER OR SENIOR ANALYST 11
5.3 QA DEPARTMENT 11

6. OPERATION PROCEDURES 12

6.1 GENERAL 12
6.2 INSTRUMENTAL DETECTION LIMITS (IDLS) 12
6.3 METHOD DETECTION LIMITS (MDLS) 14
6.4 REPORTING LIMITS 18
6.5 PRECISION AND ACCURACY (P&A) STUDIES 20
6.6 CONTROL LIMITS 22
6.7 LABORATORY UNCERTAINTY 24

7. REPORTS 24

7.1 DATA PACKAGE ORGANIZATION 24

8. REFERENCES 25

APPENDIX I 26
STUDENT'S *T* VALUES 26
APPENDIX II 27
MDL & P&A INFORMATION SHEETS 27

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) and SW846.

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 (usually annually). 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.

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. 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 are determined initially for an analysis, generally using limits supplied in the method or defined by the program (such as CLP). 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.

1.1.6 This SOP is designed for applicability to a wide variety of sample types ranging from reagent water to solids containing the 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. 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 change in a current method.

1.1.9 MDLs should be determined approximately annually for common procedures and as needed for procedures which may be performed on an infrequent basis.

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 once every year is considered impractical. However, at least one PCB/multi-component pesticide MDL must be determined annually and all of these MDL determinations must be performed within 3 years.

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

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. All MDL data are entered into the laboratory MDL database.

> **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.

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. Where no method guidance is provided, the replicates should be prepared at concentrations of 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 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.

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 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.

1.2.6 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.

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 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 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:

$$s = \sqrt{\frac{\sum\left(X_i - \overline{X}\right)^2}{n-1}}$$

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 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.

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 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.

5.1.6 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. 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 within the guidelines of the method and to perform a review of the final data prior to submission to QA. 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 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. 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.

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.

6. Operation procedures

6.1 General

6.1.1 All studies must be given laboratory LIMS ID numbers. Although they may be initially stored in QA or directly in the department, they will eventually be moved into the laboratory filing system and must have identification numbers in order to be able to retrieve the raw data. Identification numbers will almost always be assigned by QA but in the absence of the QA Officer may be assigned by authorization of QA or the Laboratory Director. Most studies will use the SAM client code QC_Officer in order to better track them at a later date. At the time of this revision, Laucks is in the process of implementing a new LIMS. The mechanics of the assigning and tracking data from the new system will change but the fundamentals of the process should not. More specific details on the new mechanics will be incorporated into a future revision when they have been developed and are reasonably solidified.

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 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.

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 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 use the signal:noise method 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 annually for each method/matrix combination for inorganic analysis and for each method/matrix/instrument combination that will be used for most organic methods and ICP/MS where there are multiple instruments.

6.3.2 MDL studies must also be performed when any major changes have been made in an instrument, such as a detector change.

6.3.3 Prior to beginning an MDL study, a laboratory ID must be obtained from QA. The data generated from the study is then referenced to that workorder or SDG in the same manner as routine sample data.

6.3.4 MDL studies must contain the following information (not necessarily in this order). This will be accomplished by using the MDL database report plus an MDL Information Sheet (See Appendix 2). The new LIMS may 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 (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
- Concentration Units
- Reporting Limits (RLs)
- Analysts signature & date signed
- Supervisor or senior analyst review signature & date signed

6.3.5 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 demonstrably lower than the RLs unless there is a specific request to report down to the MDL.

6.3.6 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

6.3.7 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

6.3.8 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.

6.3.8.1 Reagent (blank) water should be as free of analyte as possible. Reagent or interference

> **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. Although the analyst should make his/her best effort to spike at a level from 1 to 5 times the MDL, Laucks considers up to 10 times the MDL to be a sufficient concentration. Limited exceptions to this rule may be granted as long as the deviations are not great and they are approved by QA.

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.

6.3.8.2 Spiking levels which are determined to be less than 1x or greater than 10x the MDLs should in most circumstances be re-analyzed at a more appropriate spiking level.

6.3.9 Preparation of Spiked Samples

6.3.9.1 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.

6.3.9.2 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.

6.3.10 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.

6.3.10.1 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

6.3.10.2 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.

> **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, set of 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 7 points are used in the MDL determination, the current MDL database will not accommodate the calculation. In this case, the determinations will necessarily be done using a spreadsheet program.

6.3.10.3 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

6.3.10.4 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.

6.3.11 Methodology Exceptions/Specifics

6.3.11.1 Wet Chemistry

6.3.11.1.1 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.

6.3.11.1.2 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.

6.3.11.2 GC and Gas Chromatography/Mass Spectrophotometry

6.3.11.2.1 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 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.

6.3.11.2.2 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.

6.3.11.2.3 For the SIM methods this does not apply.

6.3.11.2.4 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 standard

6.4.3 The administrative decision method is generally based on what the laboratory considers to be a limit which can be obtained on a consistent and reliable basis. Values obtained from 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 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.

6.4.8 Reporting Limits are generally detailed in the Detection Limits Database and the LIMS system, unless set by project-specific agreement, in which case they are detailed in documents pertaining to that project. The only persons given the capability to edit the approved limits are QA, LIMS system administrators, and the Technical or Laboratory Director. In most cases, only QA will actually perform any such editing. 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 some cases, it may 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.

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 2) and an Excel spreadsheet with the calculated P&A values and limits. The new LIMS may 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
- 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 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 updated 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.

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.

6.6.9 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 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 workorder or 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 workorders. However, it must all be easily recoverable if full documentation is required, up until the standard laboratory data disposal date. Rationalizations for 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.

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, March 2005

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

Appendix II

MDL & P&A Information Sheets

Laucks Testing Laboratories, Inc.
MDL Information Sheet

This form must be submitted in addition to the information supplied on the SAM MDL summary form along with the supporting raw data.

MDL Database Name: _____

Workorder or 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: _____

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.

Workorder or 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: _____

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1012

Title: Solvent QC Monitoring for Trace Residue Analysis

Revision history:

Number	Date
2	4/27/99
1	4/10/91

Written by: Harry Romberg Date: 4-29-99
Harry Romberg, Quality Assurance Officer

Reviewed by: Monica Carr Date: 4/29/99
Monica Carr, Organics Division Manager

Approved by: Kathy Kreps Date: 4/29/99
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE **3**

1.1 DESCRIPTION 3

2. EQUIPMENT LIST AND REAGENTS **3**

2.1 EQUIPMENT AND REAGENTS 3

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL **3**

3.1 SAFETY PRECAUTIONS 3
3.2 WASTE DISPOSAL 3

4. OPERATION PROCEDURES **3**

4.1 SEQUESTERING OF SOLVENT 3
4.2 INITIATION, DATA HANDLING AND RECORD MAINTENANCE 4
4.3 SOLVENT ANALYSIS 5
4.4 DATA PACKAGE ORGANIZATION 7

APPENDIX I **8**

SOLVENT ACCEPTANCE CRITERIA 8

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 or freon 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 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 each week 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 workorder is created. 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 workorder. This "fraction" ID must contain the manufacturer, lot number, solvent, and tests (test codes) to be performed.

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 MECLAC, MSQCCK, and PXQCCK. 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 MECLAC, MSQCCK, and PXQCCK.

4.2.2.2 Acetone is checked for semivolatiles and pesticides/PCBs. The respective test codes assigned to these analyses are MSQCCK, and PXQCCK.

4.2.2.3 Hexane is checked only for pesticides. The respective test code assigned to this analysis is PXQCCK.

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 I. 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 QA Officer. After all of the QC in the month has been closed and signed off, the data file and acceptance sheets will be filed with the regular workorder files.

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 .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 uL of the 0.01 N NaOH. Swirl to mix well.

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 SAM report under the associated regular SAM test code, MECLAC. 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 SAM. Data and 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 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 400 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 150 mL. 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.

4.3.6 Acceptance criteria are compiled in Appendix I 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 Data Package Organization

4.4.1 A copy of the signed acceptance form along with the raw data is retained by QA under the assigned laboratory number. The Extractions supervisor keeps a photocopy of the signed approval sheet in a file in the extractions lab.

Appendix I

Solvent Acceptance Criteria

Solvent Acceptance Criteria

Semivolatile Compounds	Total ng in 1 mL.
Phenol	5000
bis(2-Chloroethyl) ether	5000
2-Chlorophenol	10000
1,3-Dichlorobenzene	5000
1,4-Dichlorobenzene	5000
1,2-Dichlorobenzene	5000
2-Methylphenol	5000
2,2'oxybis(1-Chloropropane)	5000
4-Methylphenol	5000
N-Nitroso-di-n-propylamine	5000
Hexachloroethane	5000
Nitrobenzene	5000
Isophorone	5000
2-Nitrophenol	10000
2,4-Dimethylphenol	5000
bis(2-Chloroethoxy)methane	5000
2,4-Dichlorophenol	10000
1,2,4-Trichlorobenzene	5000
Naphthalene	5000
4-Chloroaniline	5000
Hexachlorobutadiene	5000
4-Chloro-3-methylphenol	5000
2-Methylnaphthalene	5000
Hexachlorocyclopentadiene	5000
2,4,6-Trichlorophenol	10000
2,4,5-Trichlorophenol	10000
2-Chloronaphthalene	5000
2-Nitroaniline	5000
Dimethylphthalate	25000
Acenaphthylene	5000
2,6-Dinitrotoluene	5000
3-Nitroaniline	5000
Acenaphthene	5000
2,4-Dinitrophenol	10000
4-Nitrophenol	10000
Dibenzofuran	5000
2,4-Dinitrotoluene	5000
Diethylphthalate	25000

Semivolatile Compounds	Total ng in 1 mL.
4-Chlorophenyl-phenyl ether	5000
Fluorene	5000
4-Nitroaniline	10000
4,6-Dinitro-2-methylphenol	10000
N-nitrosodiphenylamine	5000
4-Bromophenyl-phenylether	5000
Hexachlorobenzene	5000
Pentachlorophenol	10000
Phenanthrene	5000
Anthracene	5000
Carbazole	5000
Di-n-butylphthalate	25000
Fluoranthene	5000
Pyrene	5000
Butylbenzylphthalate	25000
3,3'-Dichlorobenzidine	10000
Benzo(a)anthracene	5000
Chrysene	5000
bis(2-Ethylhexyl)phthalate	25000
Di-n-Octylphthalate	25000
Benzo(b)fluoranthene	5000
Benzo(k)fluoranthene	5000
Benzo(a)pyrene	5000
Indendo(1,2,3-cd)pyrene	5000
Dibenz(a,h)anthracene	5000
Benzo(g,h,i)perylene	5000

Solvent Acceptance Criteria

Pesticide/PCB Compounds	Total ng in 1 mL.
alpha-BHC	5
beta-BHC	5
delta-BHC	5
gamma-BHC	5
Heptachlor	5
Aldrin	5
Heptachlor epoxide	5
Endosulfan I	5
Dieldrin	10
4,4'-DDE	10
Endrin	10
Endosulfan II	10
4,4'-DDD	10
Endosulfan sulfate	10
4,4'-DDT	10
Methoxychlor	50
Endrin ketone	10
Endrin aldehyde	10
alpha-Chlordane	5
gamma-Chlordane	5
Toxaphene	500
Aroclor-1016	100
Aroclor-1221	200
Aroclor-1232	100
Aroclor-1242	100
Aroclor-1248	100
Aroclor-1254	100
Aroclor-1260	100

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
1	8/31/92
2	4/17/96
3	6/3/96
4	9/12/00
5	8/11/04
6	12/7/05

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

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

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 METHOD DESCRIPTION 3
1.2 DEFINITION OF TERMS 3

2. EQUIPMENT LISTS, REAGENTS AND STANDARDS 4

2.1 EQUIPMENT 4
2.2 REAGENTS 4
2.3 STANDARDS 4
2.4 COMPUTER WITH NETWORK AND iLIMS DATABASE ACCESS 5
2.5 STANDARD/REAGENT CHEMICAL LOGBOOKS – THESE LOGBOOKS ARE USUALLY ELECTRONIC 5

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 5

3.1 SAFETY PRECAUTIONS 5
3.2 WASTE DISPOSAL 5

4. OPERATION 5

4.1 REAGENT CHEMICAL PRACTICES 5
4.2 PREPARATION OF ORGANICS AND INORGANICS MATERIALS 6
4.3 TRACEABILITY DOCUMENTATION FOR ORGANIC AND INORGANIC STANDARDS 8
4.4 TRACEABILITY DOCUMENTATION FOR ORGANIC AND INORGANIC LABORATORY CONTROL SAMPLES (LCS) AND STANDARD REFERENCE MATERIALS (SRM) 18
4.5 STORAGE OF STANDARDS AND REFERENCE MATERIALS 20
4.6 SHELF LIFE 22

5. STANDARD VERIFICATION 22

5.1 CRITERIA 22

APPENDIX 1 24

EXAMPLE CALCULATIONS 24

APPENDIX 2 27

SHELF LIFE GUIDELINES 27

APPENDIX 3 29

BENCH SHEET EXAMPLES 29

APPENDIX 4 32

"HOW TO" iLIMS DOCUMENTS 32

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.

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.*

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

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.

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 pipets should be calibrated at the frequency and as described in Laucks SOP 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. 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.

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

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.

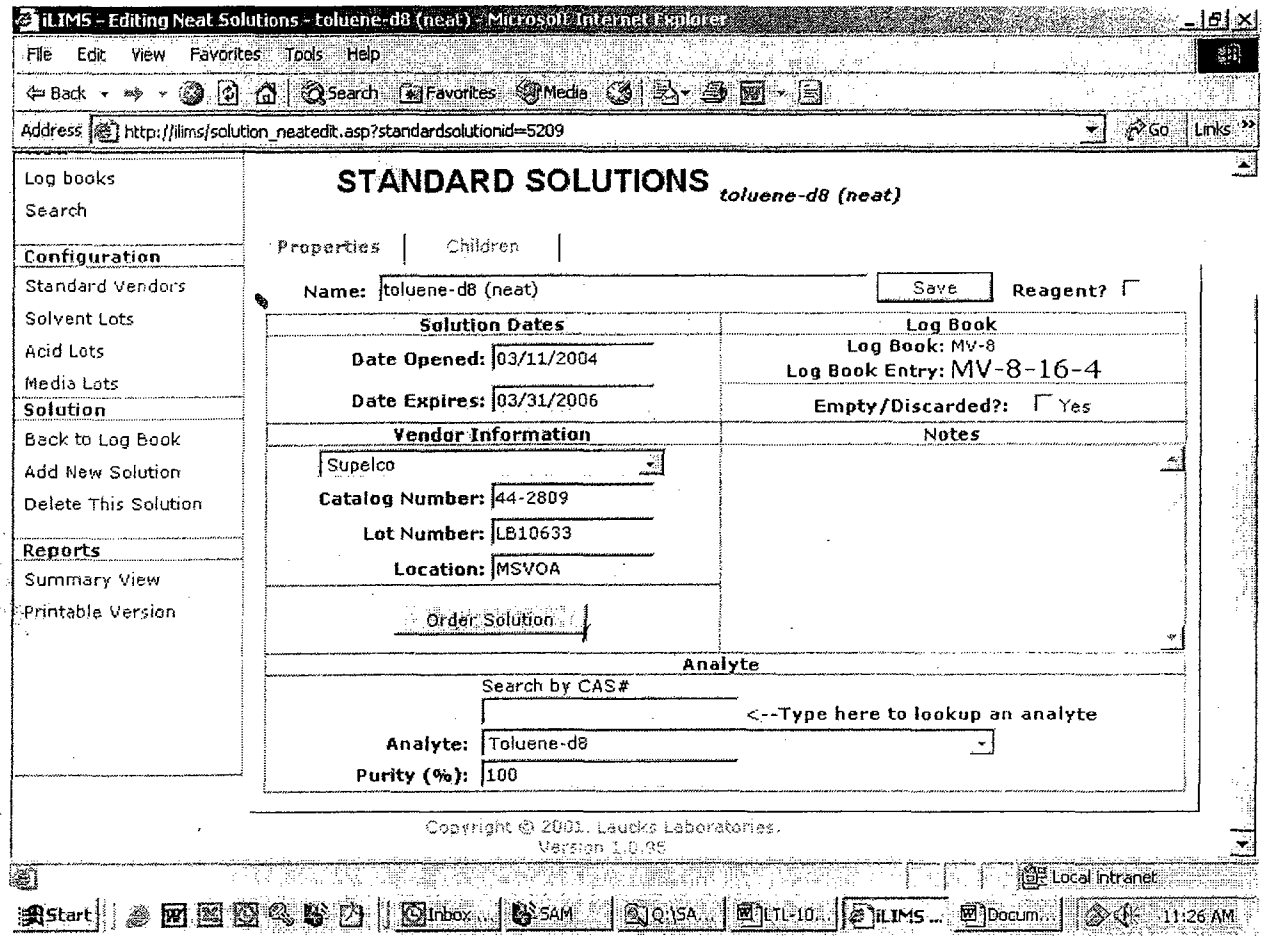
4.3.5 Materials must be consistently named so that each and every identifier/analysis for which that solution will be used are delineated (i.e. a gasoline standard must be identified as gasoline, GRO, Gasoline Range Organics, etc. as all of these terms may be used for different analysis methods, even 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.

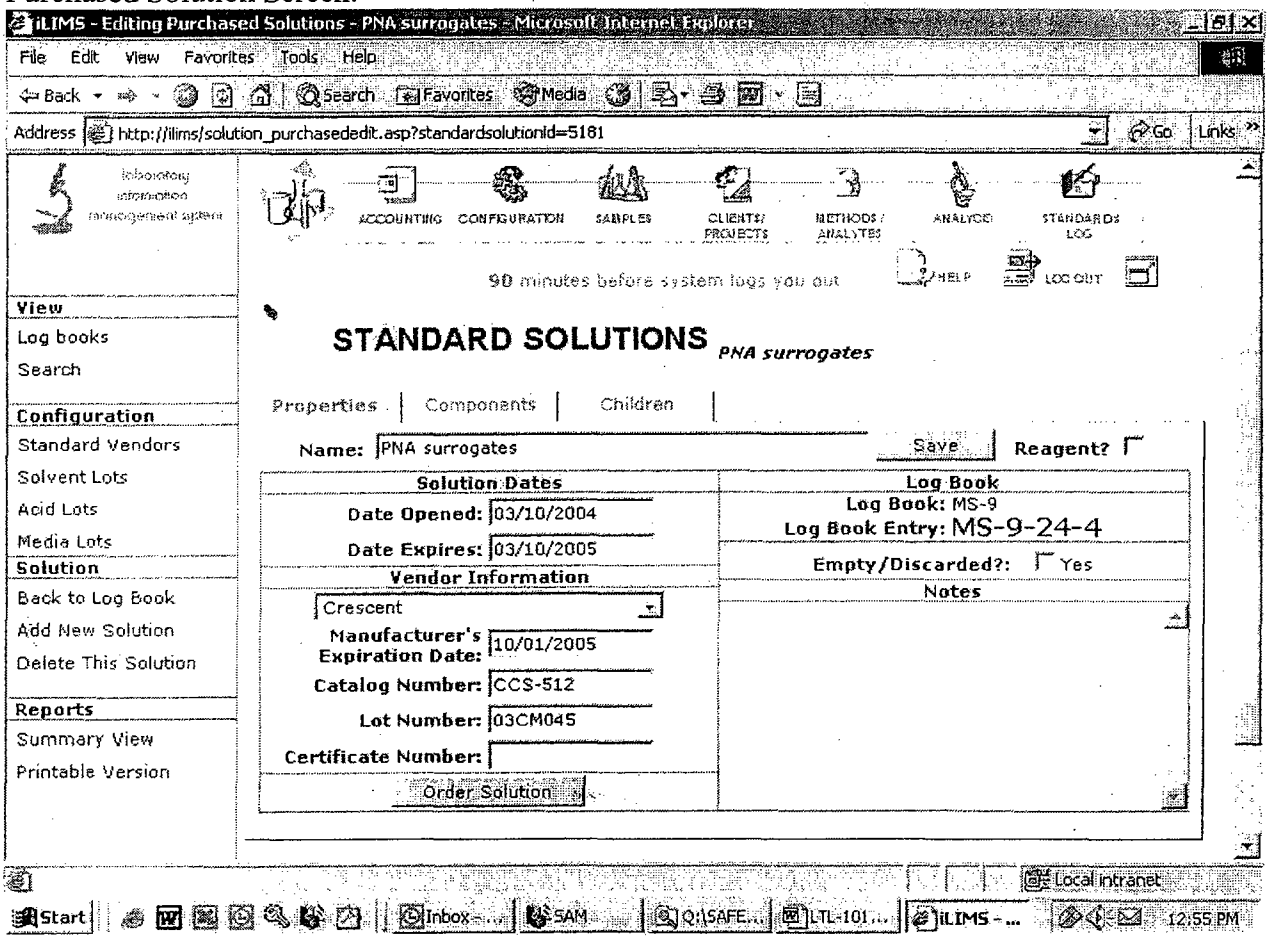
NOTE: When naming any solutions either in this section or the following section regarding Standard Reference Materials (SRMs) and Laboratory Control Samples (LCSs), it is best to use a consistent mnemonic naming system as this will greatly facilitate finding them later when they are actually used in 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.

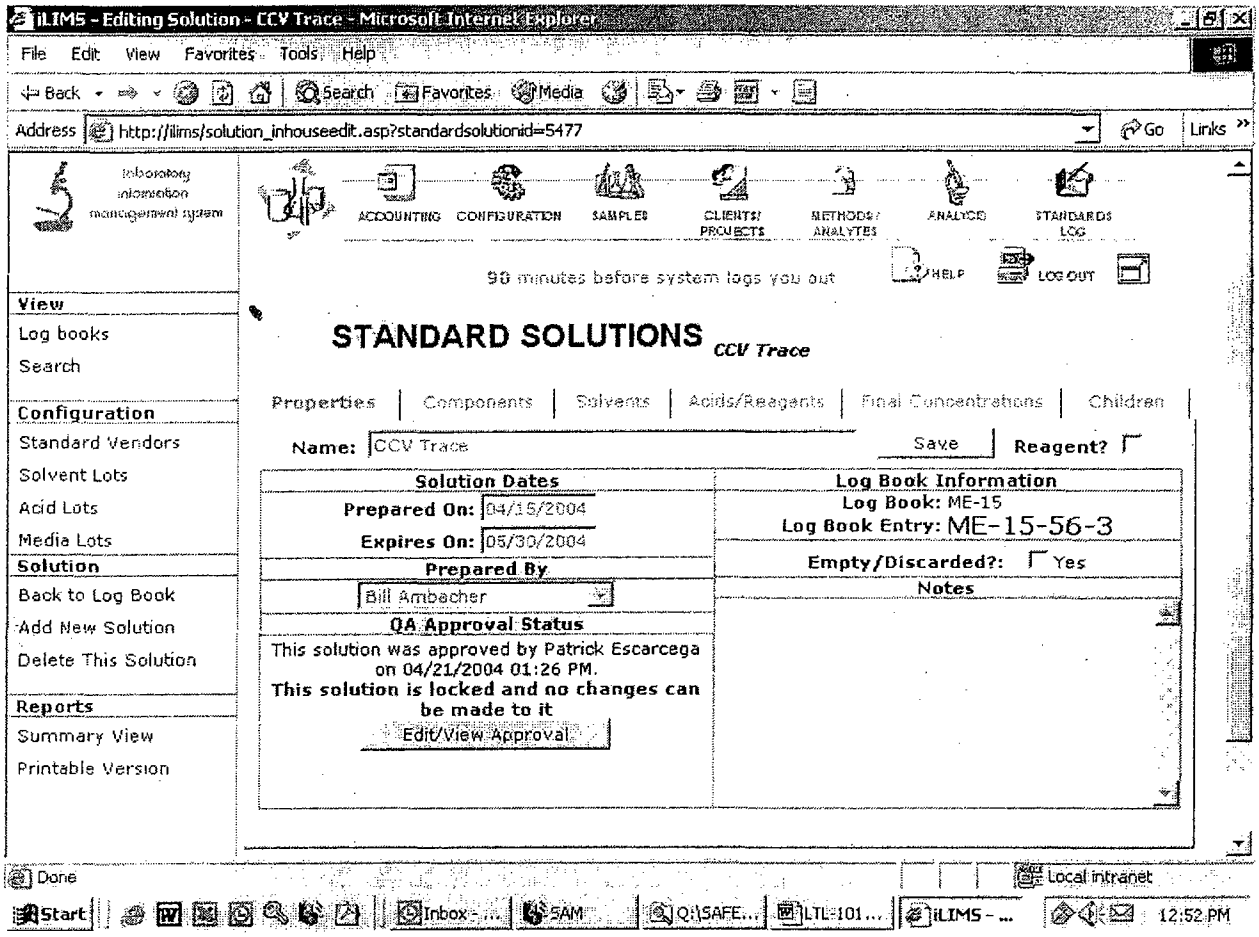
4.3.7 Neat Screen:



Purchased Solution Screen:



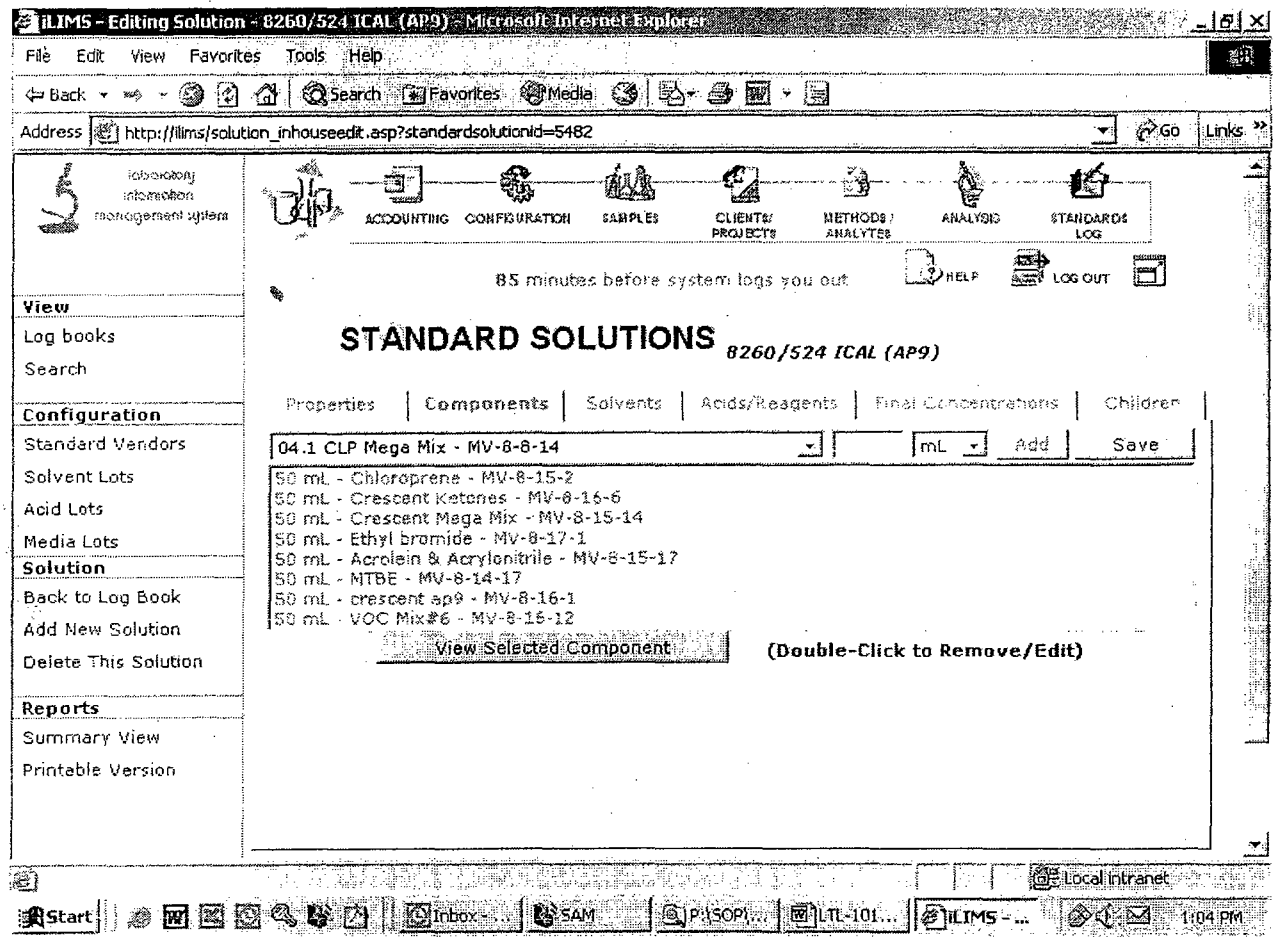
In-house Screen:



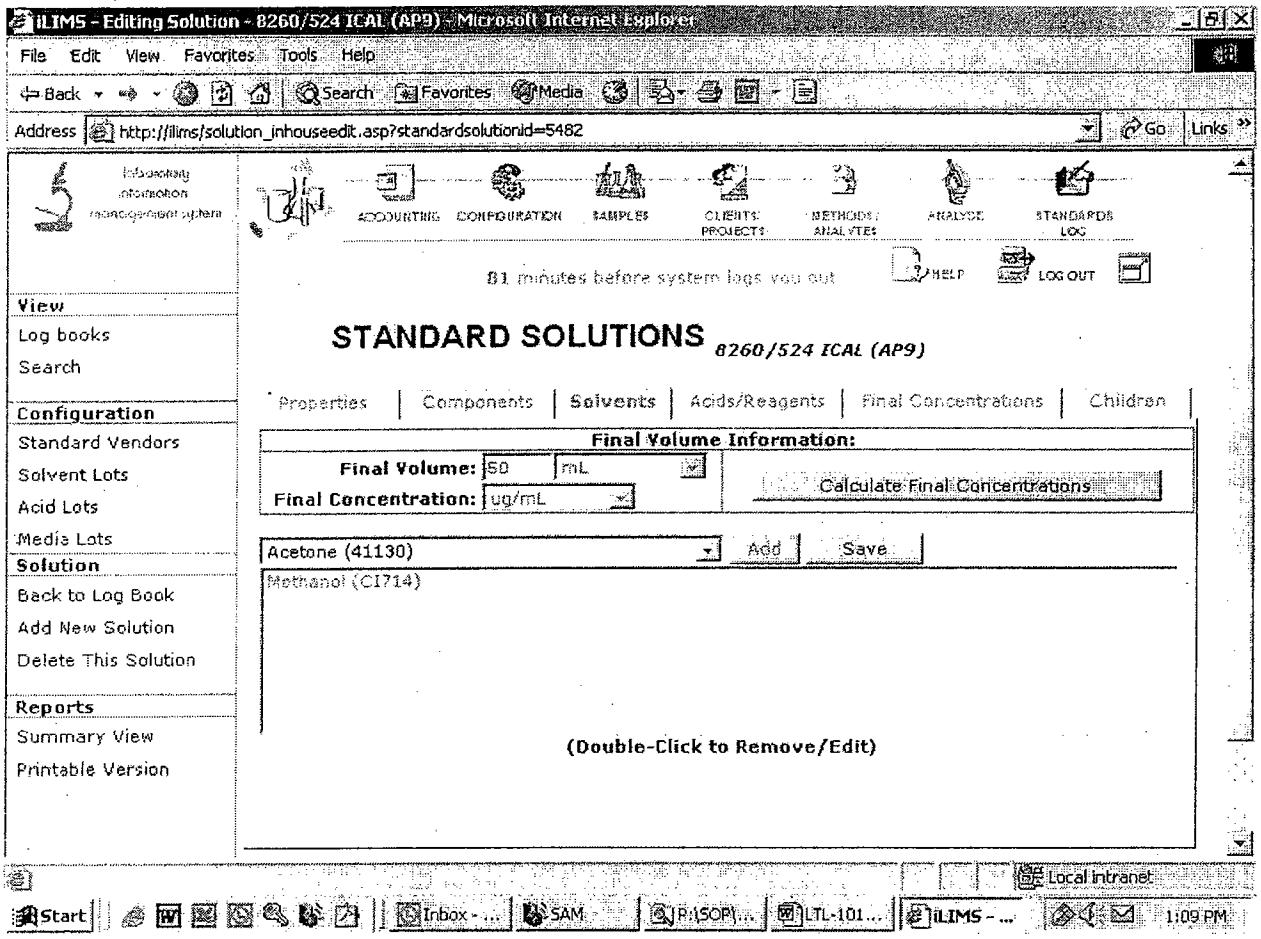
4.3.8 All elements of these property screens should be filled out as completely as possible particularly with preparation and expiration dates, vendor or preparer's names and, in the case of purchased materials, lot numbers.

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.

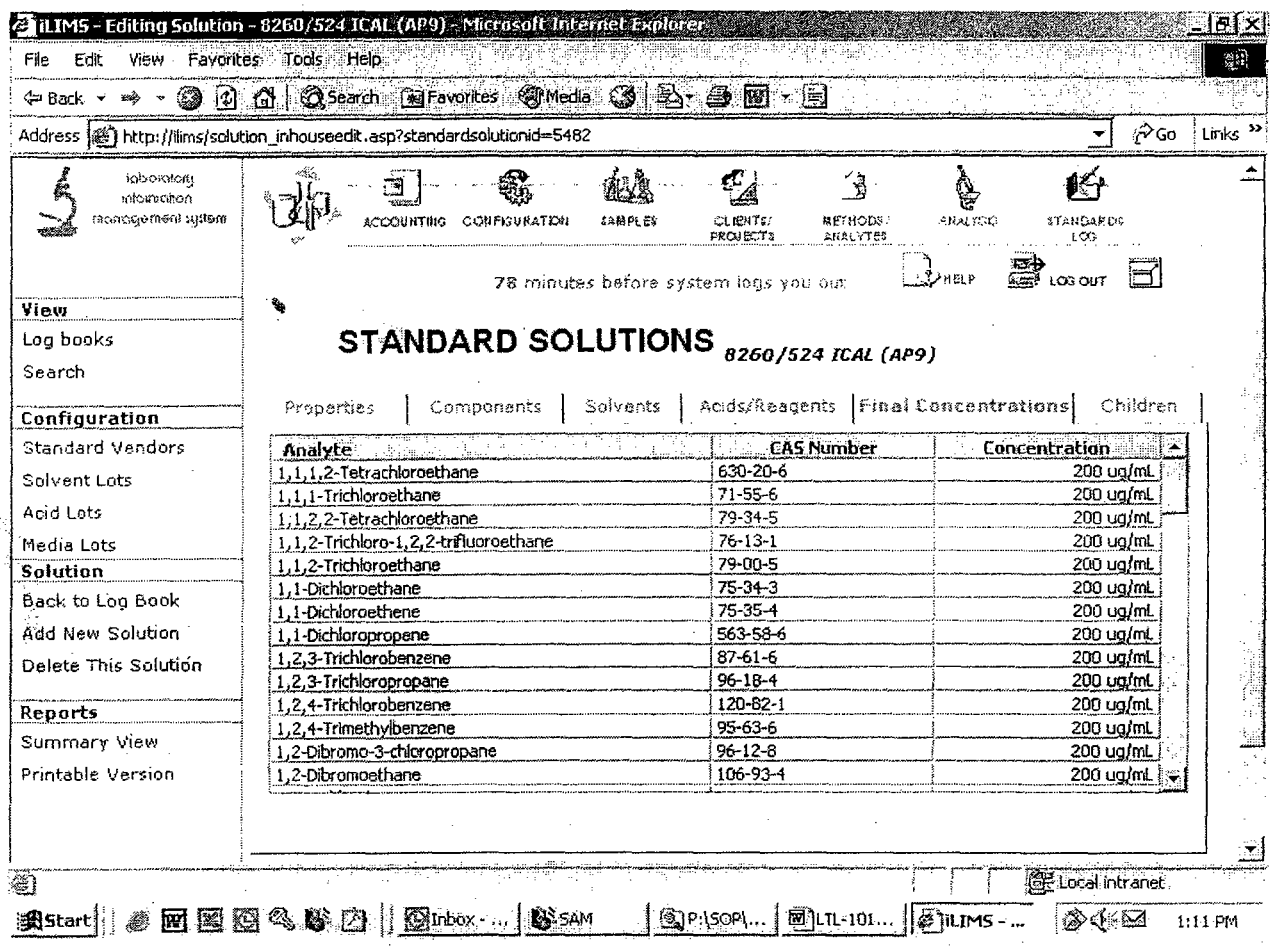
4.3.10 After one of these screens are completed, the component screen must be filled in.



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.



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

- 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.

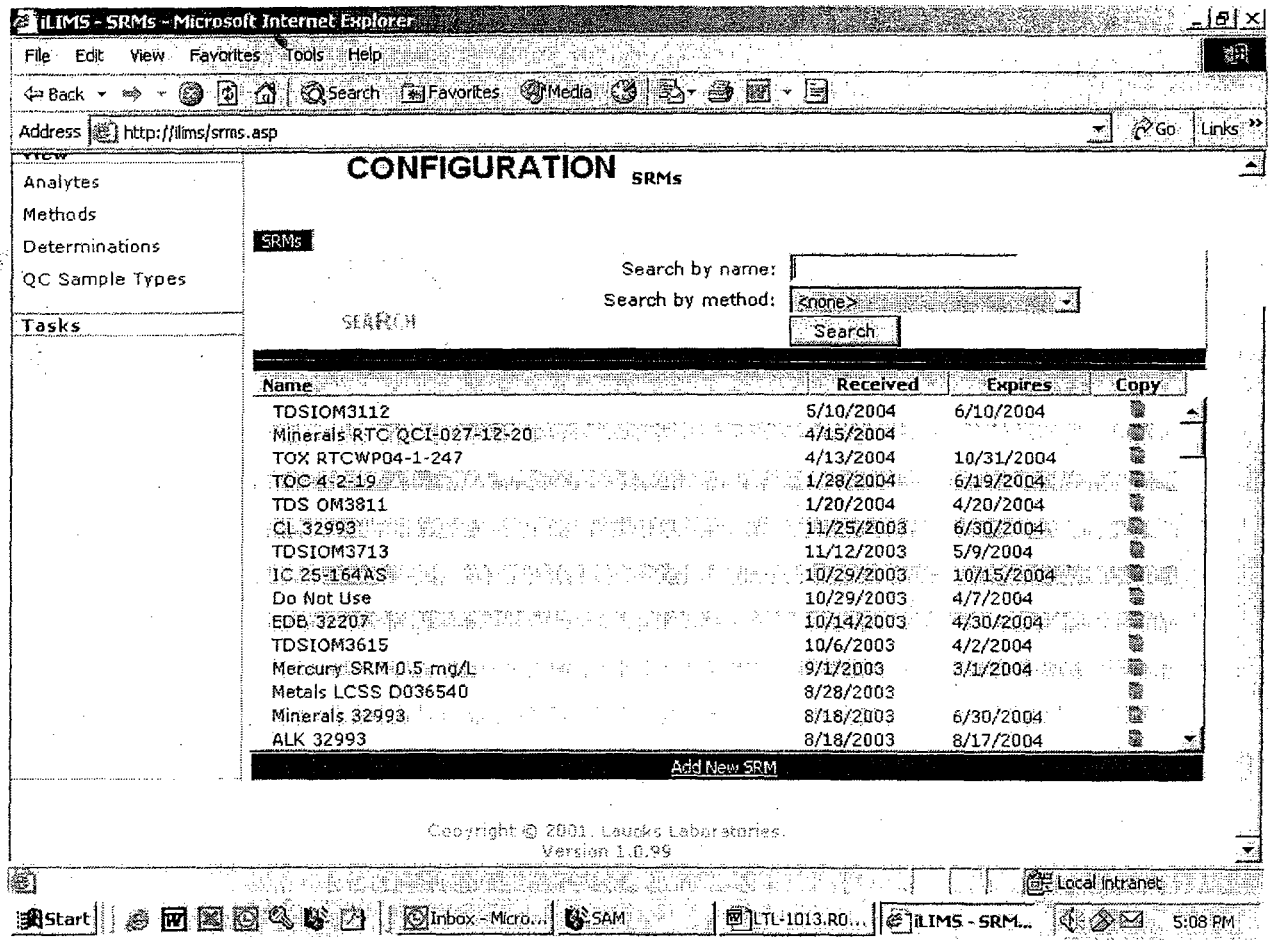
4.3.19 In addition, there are several short "How-to" documents describing how to use specific aspects of standards documentation. These are currently available by navigating to the Help screen on the iLIMS and selecting "view Howtos". Those available at the time of this writing are as follows and should be read by anyone to whom the specific situation applies. Copies of these current versions are located in Appendix 4.

- Converting Molarity or Normality Solutions to Concentration Units
- Handling Co-eluting and Degradation Compounds in the ILIMS
- Preparing Standards With Non-concentration Units
- Using the Purity Factor for Gravimetric Conversions in the iLIMS Standards Log

4.4 Traceability Documentation for Organic and Inorganic Laboratory Control Samples (LCS) and Standard Reference Materials (SRM)

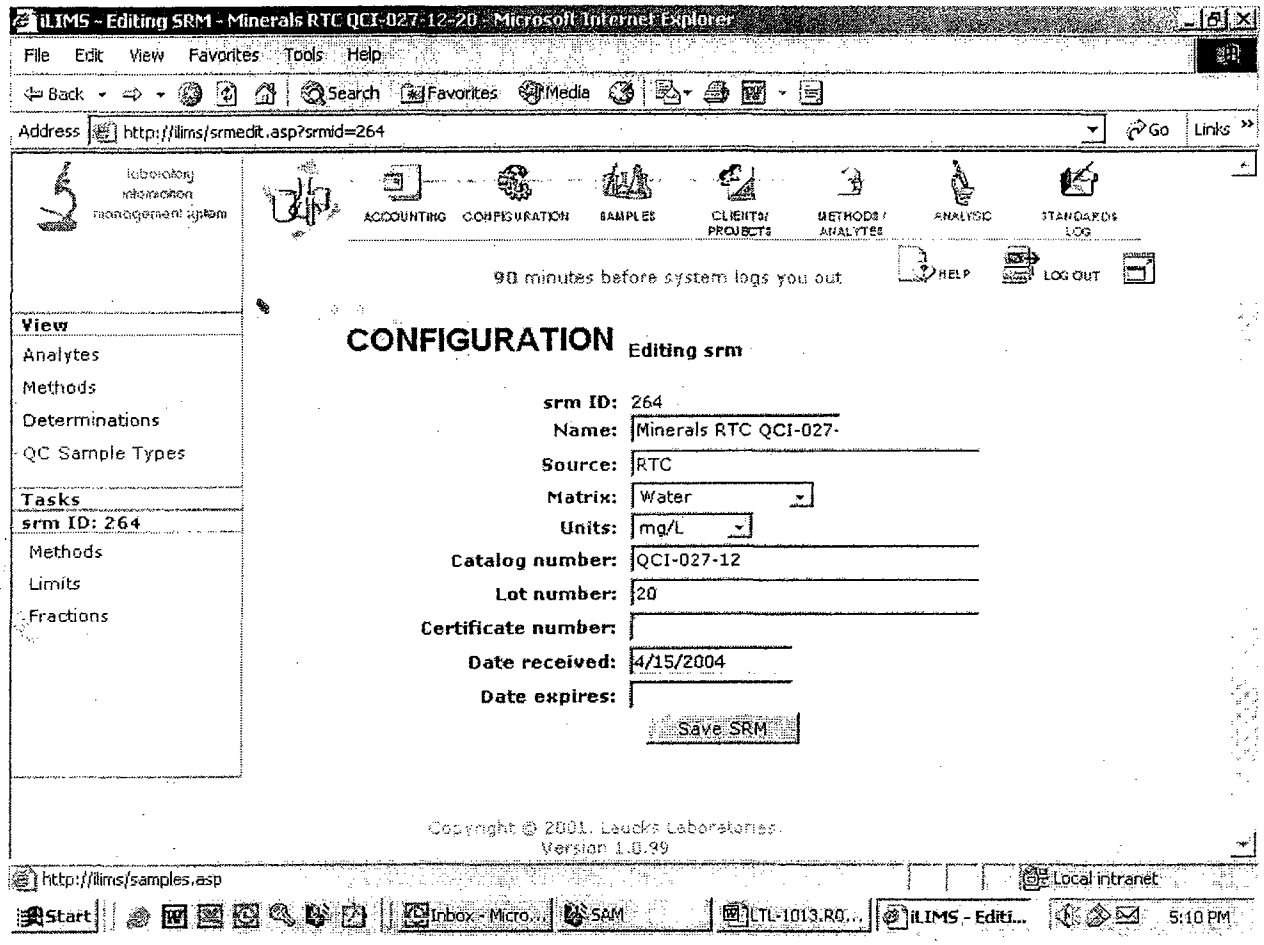
4.4.1 Only select individual supervisors or QA have access to this part of the iLIMS database. Other individuals must request one of these people to create database entries in order to make these solutions available for QC batches.

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.

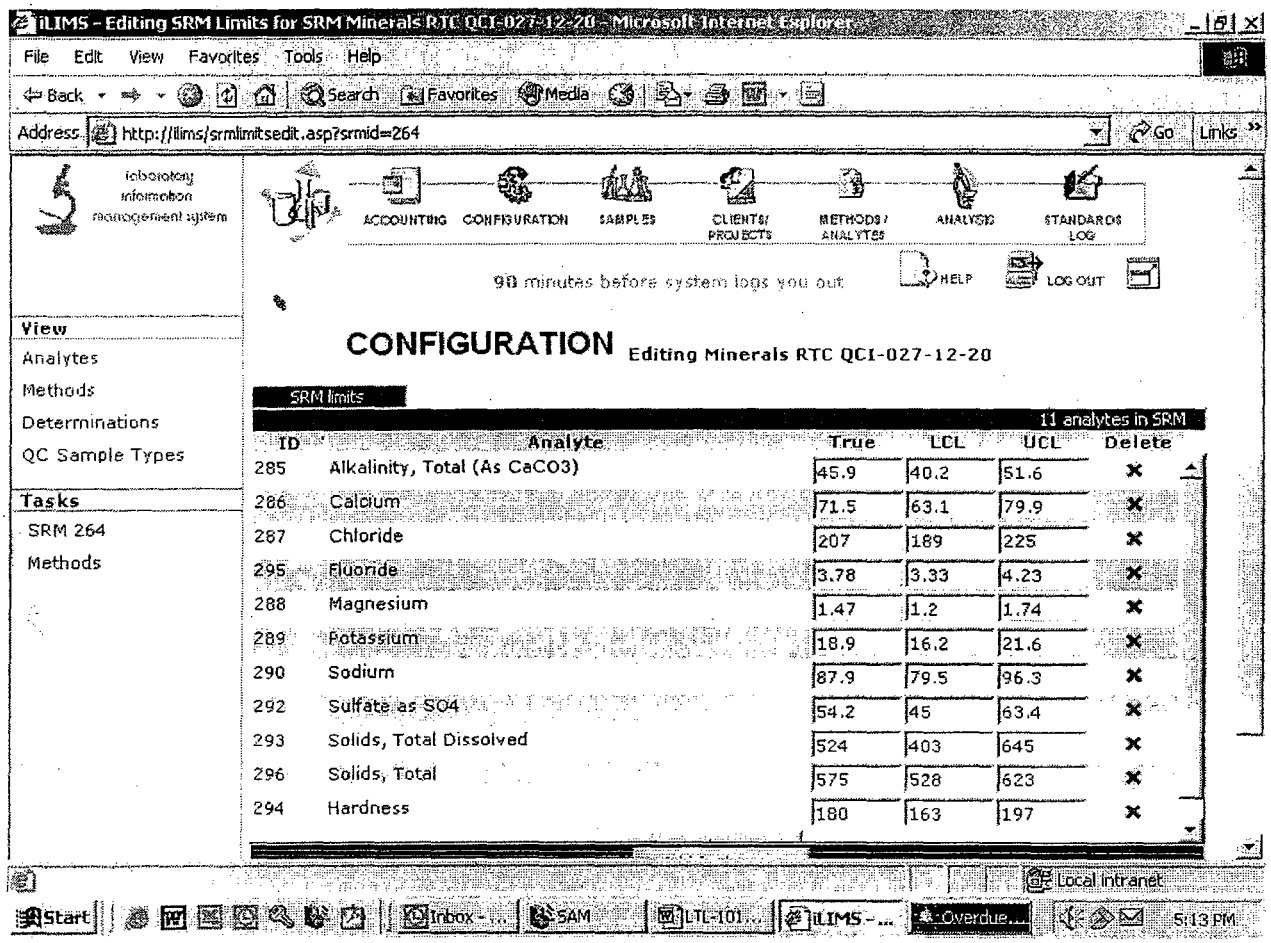
4.4.3 SRM Configuration Screen



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.

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.

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

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, 01/04, the material expires after 1/31/04. 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 5/20/04 in 1/04, instead of having a six month shelf life (07/04) the solution will expire, 05/20/04, 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

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 Subsequently, as long as the stock and child standards are logged into the iLIMS standards database and include the concentrations and amounts used in child standard preparation, it is not considered necessary to do a physical comparison of standards. However, it is still 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.

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) $$\frac{225mg}{0.01L} = 22500mg / L$$

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

$$\frac{Purity * 1{,}000{,}000 * W}{FV} = FC$$

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.

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. Gases & reactive compounds: 2 months Secondary standards: 1 month or sooner for gases & reactive compounds, e.g. styrene Other purgeables: 6 months or sooner IS, surrogate & matrix spike: fresh spiking solution weekly or sooner. Calibration standards: weekly or sooner.

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. Aqueous standards: 24 hours at 4°C or 1 hour at room temperature; 12 hours if stored on autosampler.
OLC02.1 Low Conc. CLP	Opened stock standards: weekly Aqueous standards: 24 hours. Stock Standards: 6 months or sooner Gases stock standards: 2 months or sooner Secondary dilution standards: 6 months or sooner (gases 1 month or sooner) Working calibration standards: weekly. IS: prepare fresh spiking solution weekly or sooner Surrogates: prepare fresh surrogate solution weekly or sooner
SW 846 8260B	Stock Standards: 6 months or sooner Gases: weekly if unstable or 6 months if prepared in nitrogen Working solutions: check frequently for degradation or evaporation Calibration standards are prepared daily

Appendix 3

Bench Sheet Examples

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	Analysis 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		

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

Appendix 4

"HOW TO" iLIMS Documents

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.

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**

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.

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.

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.

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

Controlled Document

No. 20 Assigned to: TeTra

Table of Contents

1. INTRODUCTION AND SCOPE **3**

1.1 METHOD DESCRIPTION 3
1.2 DISCUSSION 3
1.3 DOCUMENTATION AND FREQUENCY 3
1.4 DEFINITION OF TERMS 3

2. RESPONSIBILITIES **5**

2.1 QA PERSONNEL AND AUDIT TEAM 5
2.2 SUPERVISORS AND DEPARTMENT PERSONNEL 6

3. SAFETY PRECAUTIONS **7**

3.1 SAFETY PRECAUTIONS 7

4. OPERATION PROCEDURES **7**

4.1 GENERAL 7
4.2 AUDIT PLANNING 7
4.3 AUDIT PERFORMANCE 8
4.4 AUDIT REPORT 8
4.5 AUDIT CLOSURE AND FOLLOW-UP 9
4.6 RECORDS 10
4.7 AUDIT DISCREPANCY TRACKING 10

5. REFERENCES **12**

APPENDIX I **13**

AUDIT FINDING REPORT FORM 13

APPENDIX II **15**

FINDING TYPES 15

APPENDIX III **19**

AUDIT SCHEDULE 19

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.

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.

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

- 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.

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.

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).

- 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 <u>and to prevent recurrence</u>.

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.

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

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

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

Appendix I

Audit Finding Report Form

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:

Date Printed 3/13/03 Page 1 of 1 Revision 1, January 31, 1996

Appendix II

Finding Types

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

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

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.

Appendix III

Audit Schedule

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.

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
1	6/21/00
2	7/28/04
3	11/30/05

Written by: Harry Romberg, Quality Assurance Officer Date: 11/30/05

Approved by: Kathy Kreps, Laboratory Director Date: 12/7/05

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3
1.1 DESCRIPTION 3
2. EQUIPMENT LIST 3
2.1 EQUIPMENT 3
3. DEFINITIONS 4
4. RESPONSIBILITIES 4
4.1 ANALYST 4
4.2 PEER OR SECONDARY REVIEW 5
4.3 SUPERVISOR 6
4.4 QA REVIEW 6
4.5 PROJECT MANAGER 7
4.6 MANAGEMENT 7
5. OPERATION PROCEDURES 8
5.1 GENERAL 8
5.2 CORRECTIVE ACTION 8
5.3 ANALYST 8
5.4 PEER OR SECONDARY REVIEW 9
5.5 SUPERVISOR 9
5.6 REPORTING 9
5.7 QUALITY ASSURANCE 9
5.8 PROJECT MANAGEMENT 10
5.9 MANAGEMENT 10
6. REPORTS AND DOCUMENTATION 10
6.1 DATA REVIEW AND SIGNATURES 10
7. REFERENCES 11
APPENDIX I 12
DATA REVIEW SCHEMATIC 12

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 forms and reporting 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 but do not necessarily follow the same process or the same level of documentation. 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 Access to computer programs, etc. which may be required in order to complete the review process

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.
- 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.
- 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.
- The analyst will either transcribe the data into the LIMS 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 (both conventional chemistry and metals), the complete data package is assembled by a data administration assistant. This assistant 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, usually not the supervisor. 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:

- 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 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).

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 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. 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 Data review forms are provided in individual data review SOPs.

6.1.2 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.

6.1.3 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

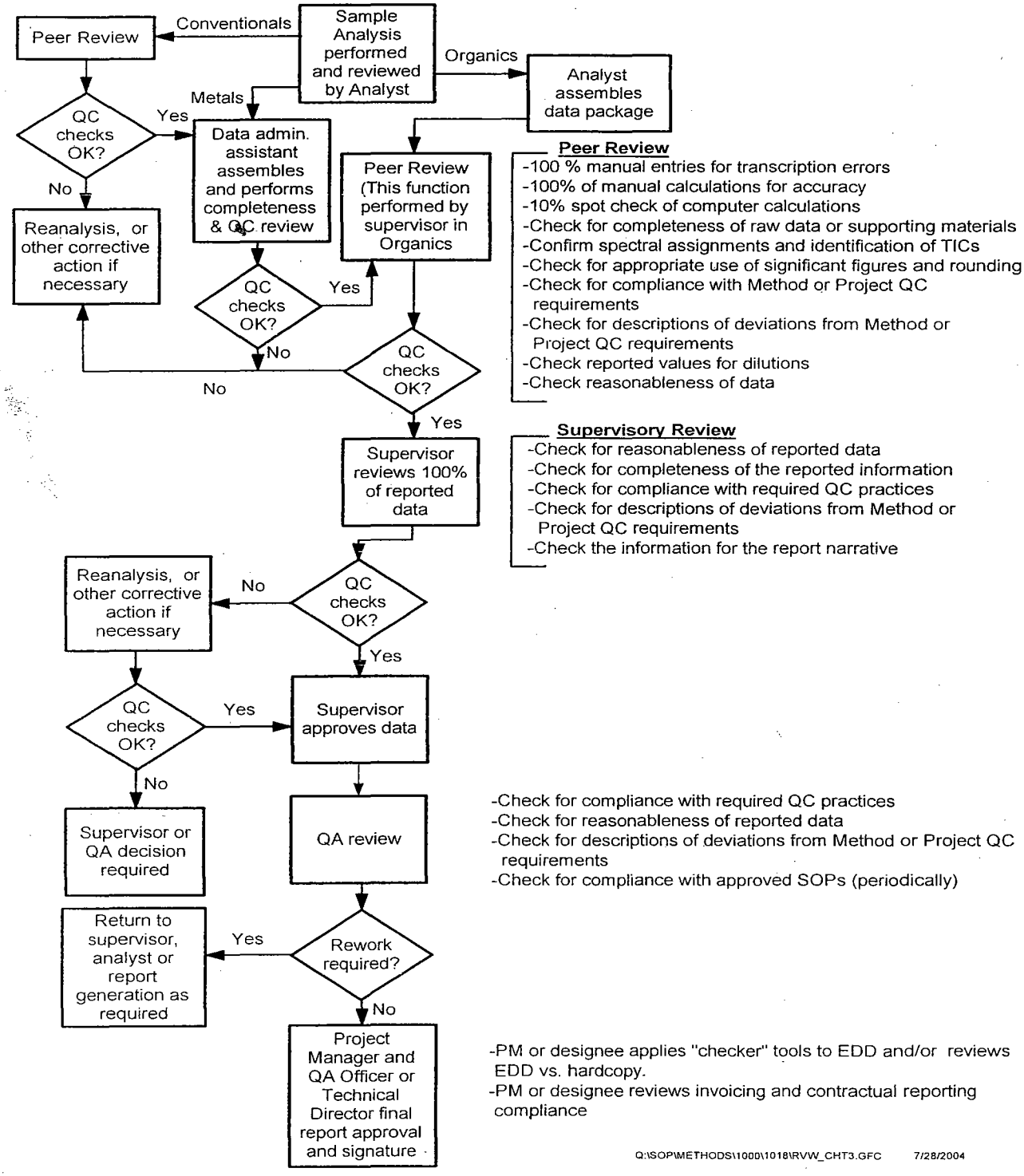
Laucks SOPs

LTL-7001	Metals Data Review
LTL-7002	EPA Inorganics 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-8201	GC/MS Volatiles Data Review
LTL-8202	GC/MS Semivolatiles Data Review
LTL-8301	HPLC Aromatics 8310 Data Review
LTL-8302	HPLC Ordnance 8330 Data Review
LTL-9001	Inorganic Conventionals Data Review

Appendix I

Data Review Schematic

Data Review and Approval



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 Date: 11/30/05
Harry Romberg, QA Officer

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

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1 SCOPE 3
1.2 PURPOSE 3
1.3 DEFINITION OF TERMS 3

2. REQUIREMENTS 3

2.1 CONTROL 3
2.2 MAINTENANCE 4
2.3 MONITORING 4

3. RESPONSIBILITIES 5

3.1 QUALITY ASSURANCE MANAGER 5
3.2 RESPONSIBLE SUPERVISOR 5
3.3 ANALYST 5

4. PROCEDURE 6

4.1 NOTEBOOK STRUCTURE 6
4.2 CONTROL OF LOGBOOKS 7
4.3 USE OF LABORATORY LOGBOOKS 8
4.4 SUPERVISORY MONITORING OF LABORATORY LOGBOOKS 9
4.5 QA MONITORING OF LABORATORY LOGBOOKS 9

APPENDIX I 10

EXAMPLE QA LOGBOOK LABEL 10

APPENDIX II 11

EXAMPLE LOGBOOK REVIEW ITEMS TO BE OBSERVED 11

APPENDIX III 12

EXAMPLE LOGBOOK REVIEW LABEL 12

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.

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.

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.

- 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.

- 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.

- 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.**

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.

Appendix I

Example QA Logbook Label

Laucks
Testing Laboratories, Inc.

QA Book No.: _____

Issued To: _____

Used For: _____

Dept. Book No.: _____

Start Date: _____

End Date: _____

Appendix II

Example Logbook Review Items to be Observed

- Have all pertinent fields been filled or marked not applicable (N/A)?
- Has empty space been crossed out properly initialed and dated?
- Have errors been corrected with single-line crossouts, initialed and dated (no obliterations or overwrites)?
- Are all entries clear and easy to read and comprehend?
- If calculations are involved, check several random calculations for error.
- If traceability is involved (as for standards) check several random entries to confirm that the logbook entries can be tracked back to the original entry.
- If standards log, observe some actual standards and compare them against logbook entries for accuracy.
- Are all handwritten entries initialed and dated?
- If the book is beginning to deteriorate, it should be repaired or retired and replaced before it completely falls apart.

Appendix III

Example Logbook Review Label

Logbook pages _____ through _____
have been reviewed for completeness and
spot-checked for accuracy.

Initials: Date:

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-1020

Title: **The Integration of IC, GC, HPLC, and GC/MS Peaks**

Revision history:

Number	Date
0	08/26/96
1	08/21/97
2	05/02/02

Revised by: Monica Carr, Organics Division Manger Date: 5/9/02

Reviewed by: Harry Romberg, QA Officer Date: 5-9-02

Approved by: Kathy Kreps, Laboratory Director Date: 5/9/02

Controlled Document
No. 20 Assigned to: TeTr

Table of Contents

1. INTRODUCTION AND SCOPE **3**

1.1 METHOD DESCRIPTION 3
1.2 DEFINITION OF TERMS 3

2. SOFTWARE **3**

3. RESPONSIBILITIES **4**

3.1 ANALYST 4
3.2 SUPERVISOR 4

4. OPERATION PROCEDURES **4**

4.1 INTEGRATOR OPERATION 4

5. DOCUMENTATION **6**

6. REFERENCES **7**

APPENDIX I **8**

FIG 1. 9
FIG 2. 10
FIG 3. 11

APPENDIX II **12**

ATTACHMENT 1 13
ATTACHMENT 2 14
ATTACHMENT 3 15
ATTACHMENT 4 16
ATTACHMENT 5 17
ATTACHMENT 6 18
ATTACHMENT 7 19
ATTACHMENT 8 20
ATTACHMENT 9 21

1. Introduction and Scope

1.1 Method Description

1.1.1 This SOP describes the integration process for chromatographic data, the procedures for manual integration, and the procedures for documenting manual integration.

1.1.2 Integration identifies peaks found in the data collected during data acquisition and characterizes them. The software uses the integrated peaks to determine the identity and quantity of compounds in the samples. The peak area, peak height, peak type, baseline, and retention time of each peak in a chromatogram are determined by integration. It is expected that situations will arise when the automated quantitation provide inappropriate quantitations. This normally occurs when there is compound co-elution, baseline noise, or matrix interference. Some peaks, due to limitations of the software, will need to be manually integrated. The manual integration process must be documented as outlined in section 5 of this SOP.

1.1.3 Manual integrations have been a source of serious concerns throughout the laboratory industry as a tool of fraud. There have been labs as well as individuals who have been penalized due to improper manual integration, or inadequate documentation that their actions were appropriate. Therefore, it is imperative that proper and honest and completely documented procedures are followed.

1.1.4 This method is restricted to use by, or under the supervision of analysts experienced in the technique described. Each analyst performing this method must have demonstrated the ability to perform the described analysis.

1.2 Definition of Terms

1.2.1 Manual Integration - any intervention by an analyst or supervisor to change the peak area, peak height, baseline, peak type, or retention time of a chromatographic peak.

2. Software

2.1.1 GC Acquisition-HP/EZChrom
2.1.2 GC/MS-Teknivent/EnviroQuant/Chemstation
2.1.3 GC-Target

3. Responsibilities

3.1 Analyst

3.1.1 The analyst is responsible for reading and understanding this SOP and that which is applicable to the method of analysis. The analyst must also perform and document all manual integrations as specified in this SOP.

3.2 Supervisor

3.2.1 The supervisor or designated data reviewer must verify that all manual integrations are performed according to this SOP. The supervisor or designated data reviewer must document that this verification has occurred per the applicable Data Review, Validation, and Reporting SOP.

4. Operation Procedures

4.1 Integrator operation

4.1.1 The ideal chromatogram has perfectly symmetric peaks separated from each other with periodic baseline points. It is common to encounter split peaks, deformed peaks, merged peaks, sloping baselines, noise, spikes, shoulders on peaks, and a host of other calamities.

4.1.2 To maximize the chances of obtaining ideal chromatograms, first optimize the chromatography.

4.1.3 Peak recognition and integration sequence - As the integrator scans the data, it examines the slope (vertical distance between points) and curvature (positive or negative). So long as these remain within preset bounds data is interpreted as the baseline. If the bounds are exceeded, a peak may be starting. If the condition persists, the integrator decides that it is on the upslope of a peak.

4.1.4 The curvature changes to negative about halfway up the peak. This is the inflection point where the peak starts to round over approaching the apex. Passing the top, the slope becomes negative and the integrator is on the downslope. Another inflection point comes on the downslope and finally the peak returns to the baseline (Figure 1).

4.1.5 Finite width of integration slices - The slope changes from positive to negative at the top of the peak. However for area slices having finite width the integrator can only determine which slice contains the peak apex. To get better values for the retention time and peak height, the

integrator takes the slice containing the-apex and one slice on either side, fits them to a quadratic equation, and solves the equation to find the highest point (Figure 2).

4.1.6 Optimizing peak recognition - The best conditions for recognizing isolated symmetric peaks on a quiet baseline is to match the peak width parameter to the measured width of the peaks at half height. Threshold should be a few units less than the highest value still capable of detecting the peak. When peaks cluster together or the baseline slopes or is noisy, these ideal values must be modified. Figure 3 shows the effects of changing the values.

4.1.7 Manual integration - It is important that the analyst is familiar with the compounds that are routinely analyzed. Knowing the response and peak shape of the standard is important for consistency in integration. It is best to optimize the method to process data so that manual intervention is minimized and peak integration is more consistent. If manual integration is performed, it is important to be consistent for a given analyte in the standards, blanks, spikes, and samples.

4.1.8 Allowable manual integrations - Some common reasons for manual integration are:

4.1.8.1 Split peaks (attachment 1)

4.1.8.2 Tailing (attachment 2)

4.1.8.3 Retention time shifts (attachment 3)

4.1.8.4 Mis-identification (attachment 3)

4.1.8.5 Merged peaks (attachment 3)

4.1.8.6 Secondary ions or qualifier ions (attachment 4)

4.1.8.7 Baseline shifts (attachment 5)

4.1.8.8 Skimming versus dropped baseline (attachment 6)

4.1.9 Improper manual integrations - These practices are not allowed and warnings up to and including termination of employment will follow any documented cases of improper manual integration. If you are unsure about a manual integration ask your supervisor or QA.

4.1.9.1 Adding area by including other peaks. (attachment 7)

4.1.9.2 Improper baseline - this includes the practice of having the baseline moved up the side of a peak to decrease the area. (attachment 8)

4.1.9.3 Changing a proper integration to make the peak "in".

4.1.10 Special rules for fuel analyses (*e.g.*, gasoline, diesel) - The integration of these multi-component analytes requires special integration rules. The area of all peaks (with the exception of the surrogates) and the area of the non-resolved components (hump) are grouped together to determine the quantity of the analyte. The baseline is fixed at the start and held at a constant level for the entire run. When integration of the baseline for fuel analyses is performed, the quantitation report will not be dated and initialed by the analyst. Instead an explanation of the baseline integration will be documented on the quantitation report or in the appropriate SOP and discussed in the sample narrative. Some acceptable multi-analyte integrations are shown in attachment 9.

5. Documentation

5.1.1 When any manual integration is performed, a graphic copy of the peak with the integration marks is generated and put into the folder with the chromatograms and quantitation reports for that sample. The three different software systems used in the laboratory are listed below with the commands for generating the copy. On the quantitation report the analyst must initial, date, and give a brief description of the reason for the manual integration (table 1). In addition, the analyst must provide chromatograms from both before and after the manual integration. The manual integration must also be documented in the associated sample narrative. The supervisor or his/her designee will look at each manual integration during data review and complete the summary on the data review or QC checklist.

5.1.2 Target: After changing the integration, exit and save in Target Review.

5.1.3 GC/MS Teknivent EnviroQuant/Chemstation: After using Qedit to change the peak click on 'Graphics Report to Printer'

5.1.4 EZChrom: After changing the integration, toggle the "Reanalyze" key and print.

Table 1

Manual Integration Key

M = Manual integration due to missed peak or irregular peak shape.
MS = Manual integration due to split peak.
MR = Manual integration due to retention time shift.
MI = Manual integration of correct isomer.
MT = Manual integration due to peak tailing.

MB = Manual integration due to irregular baseline.

6. REFERENCES

6.1.1 Target Manual, Thru-Put Systems, Inc., Target Compound Analysis Software User's Guide, 1994.

6.1.2 Envirolink User's Manual, TEKNIVENT, 1994

6.1.3 HP Chemstation, Version B.01.00

6.1.4 EZChrom Chromatography Data System, Scientific Software, Rev. 6.6, 1995.

6.1.5 Wisconsin DNR Newsletter, October 1994.

6.1.6 SOP# LTL-1018, Standard Operating Procedure for Review and Approval Practices for Validatable Packages.

6.1.7 SOP # LTL-8005, Standard Operating Procedure for GC Gas/BTEX Data Review.

6.1.8 SOP #LTL-8004, Standard Operating Procedure for GC Volatiles Data Review.

6.1.9 SOP #LTL-8001, Standard Operating Procedure for GC Hydrocarbons Data Review.

6.1.10 SOP #LTL-8301, Standard Operating Procedure for HPLC Aromatics 8310 Data Review.

6.1.11 SOP #LTL-8302, Standard Operating Procedure for HPLC Ordnance 8330 Data Review.

6.1.12 SOP #LTL-8201, Standard Operating Procedure for GC/MS VOA Data Review.

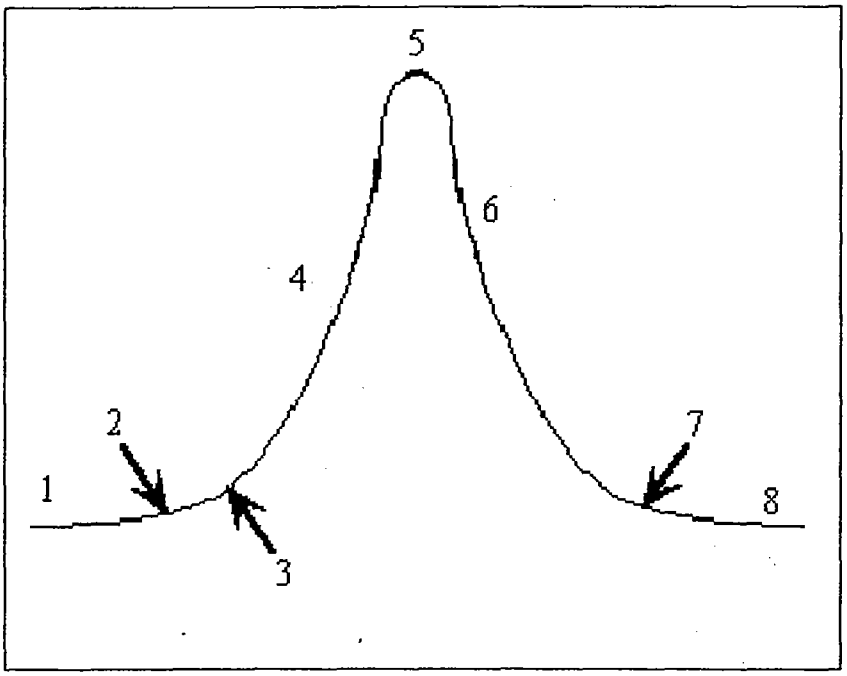
6.1.13 SOP #LTL-8202, Standard Operating Procedure for GC/MS Semivolatile Data Review.

Appendix I

Fig 1.

The sequence for finding a positive peak is:

1. Slope and curvature within limits	track baseline
2. Slope and curvature above limits	perhaps a peak?
3. Slope remains above limit	here's a peak!
4. Curvature becomes negative	front inflection point
5. Slope becomes negative	top of peak
6. Curvature becomes positive	rear inflection point
7. Slope and curvature within limits	approaching end of peak
8. Slope and curvature remain within limits	end peak, track baseline



Steps 3,5, and 8 define Cardinal Points, which are the Start of Peak, Apex, and End of Peak Respectively

Fig 2.

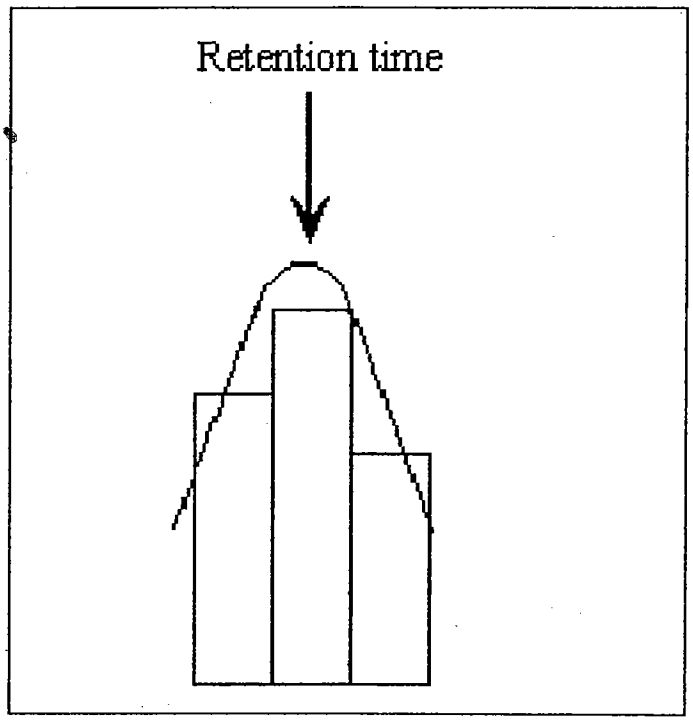
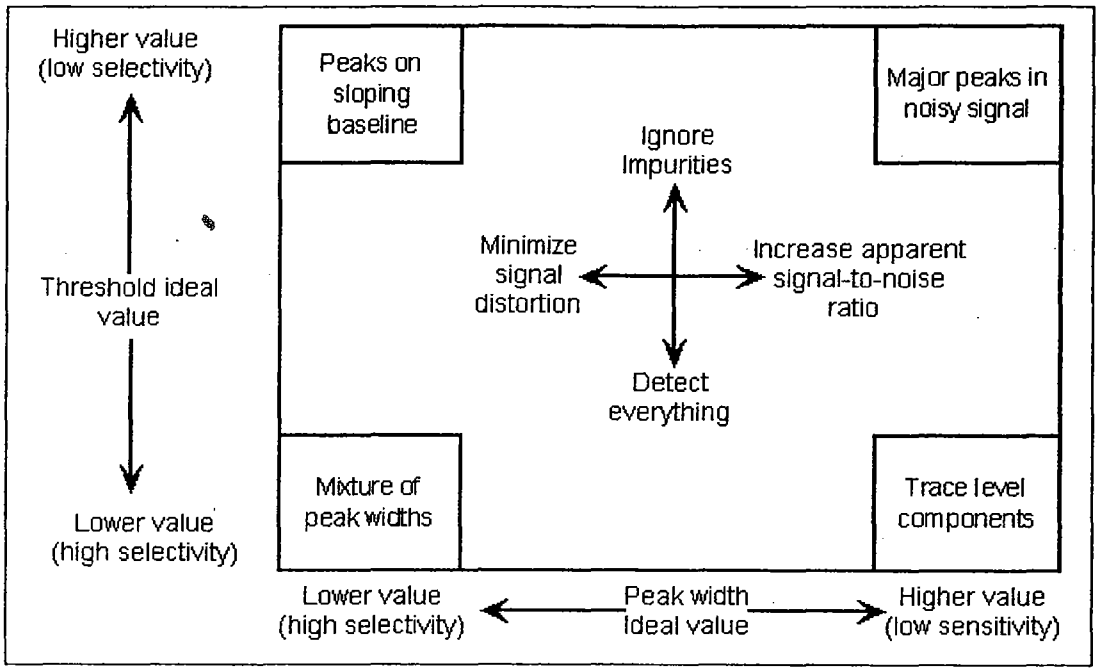
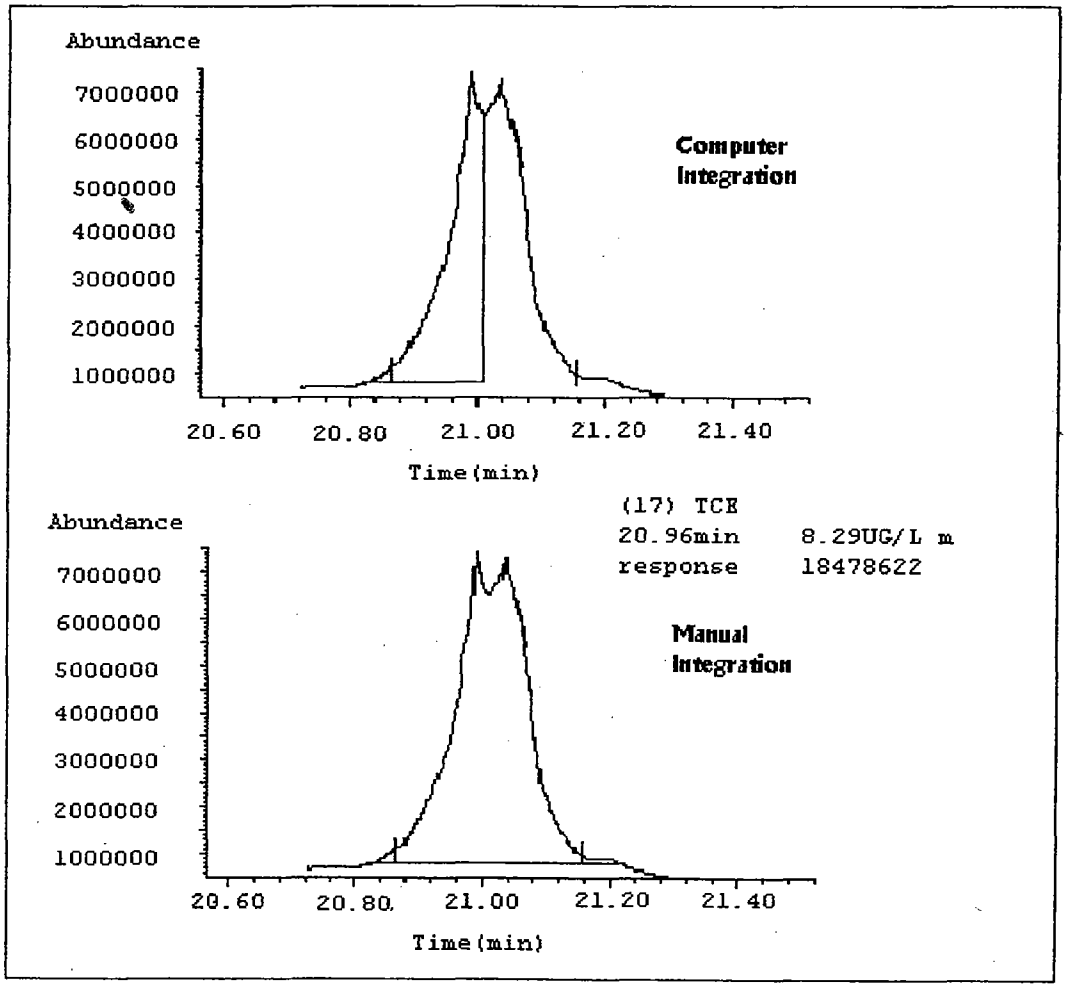


Fig 3.


Higher value
(low selectivity)
Threshold ideal
value
Lower value
(high selectivity)
Peaks on
sloping
baseline
Major peaks in
noisy signal
Ignore
Impurities
Minimize
signal
distortion
Increase apparent
signal-to-noise
ratio
Detect
everything
Mixture of
peak widths
Trace level
components
Lower value
(high selectivity)
Peak width
Ideal value
Higher value
(low sensitivity)

Appendix II

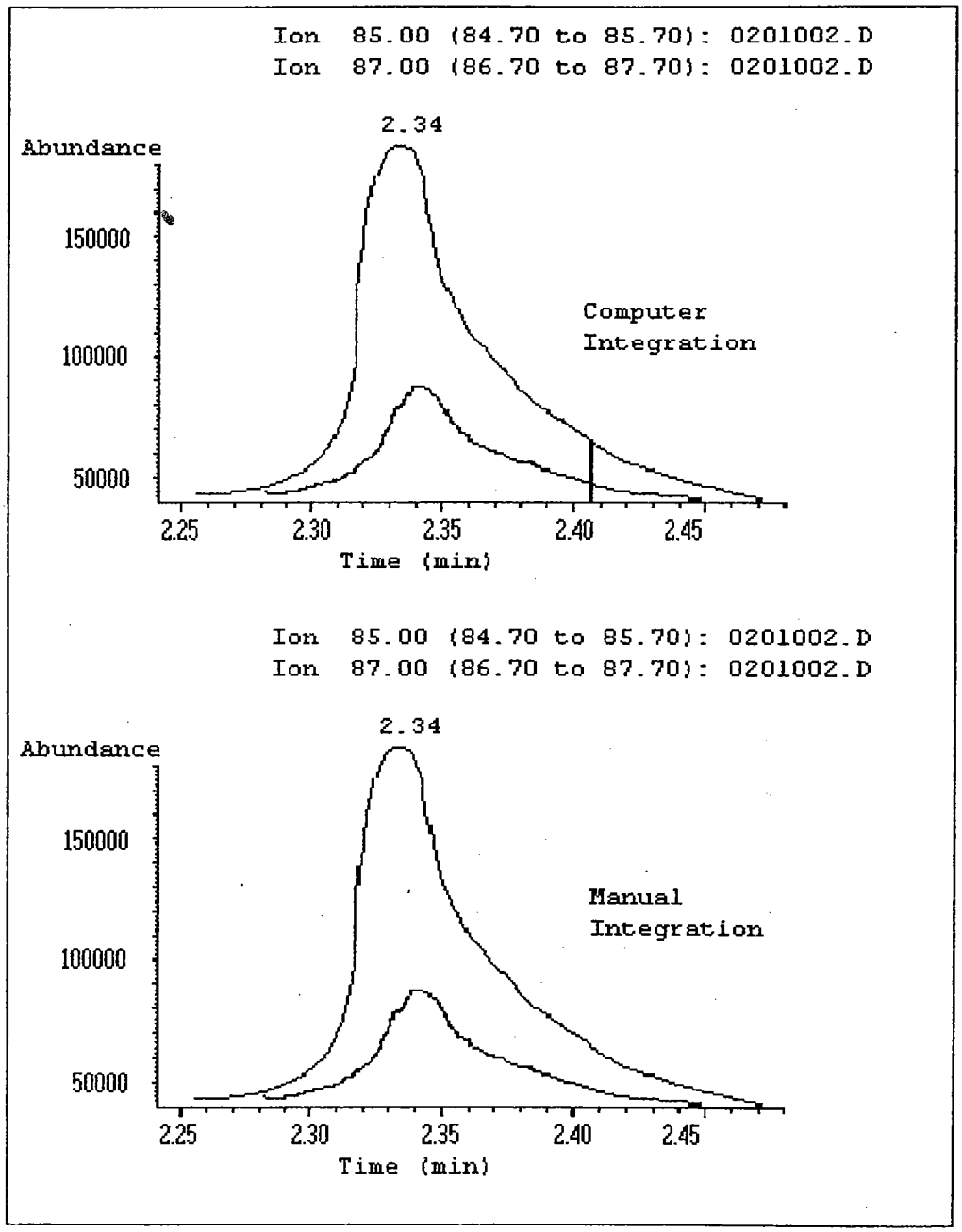
Attachment 1



Allowable Integration

Split Peaks

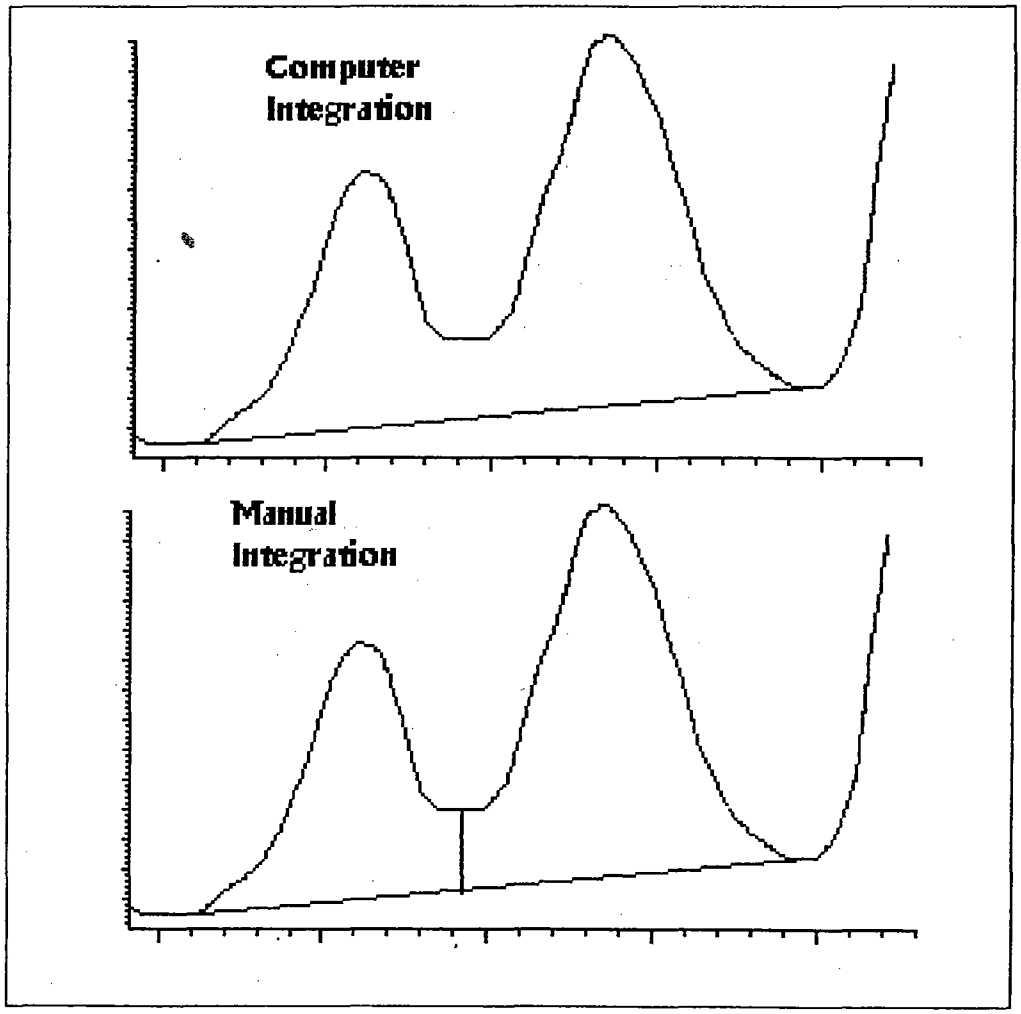
Attachment 2



Allowable Integration

Tailing

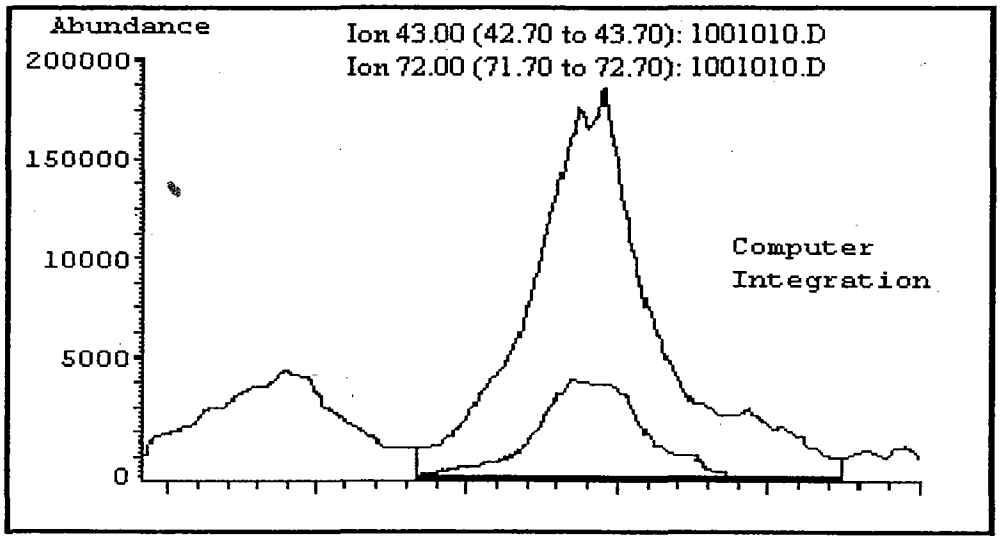
Attachment 3

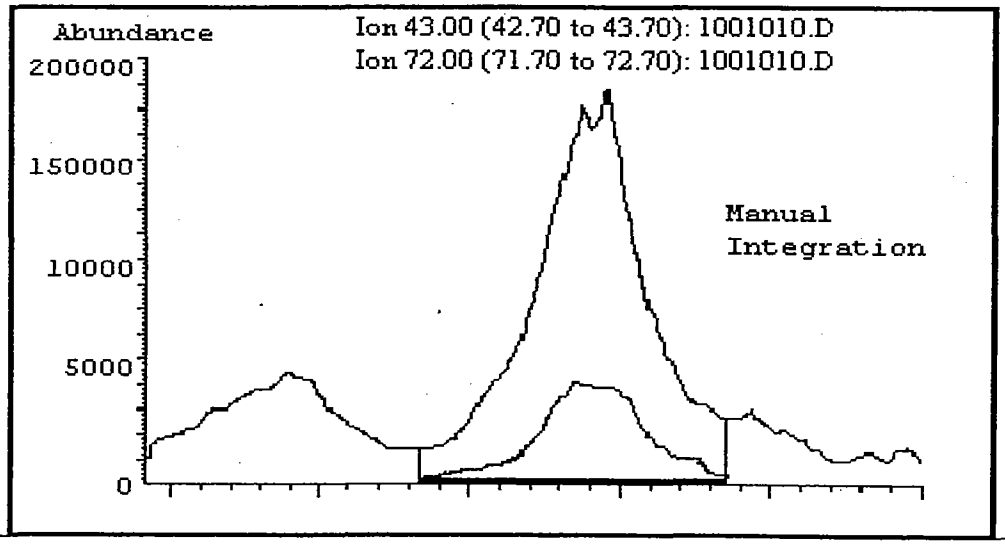


Allowable Integration

Retention Time Shift - Mis-Identification - Merged

Attachment 4

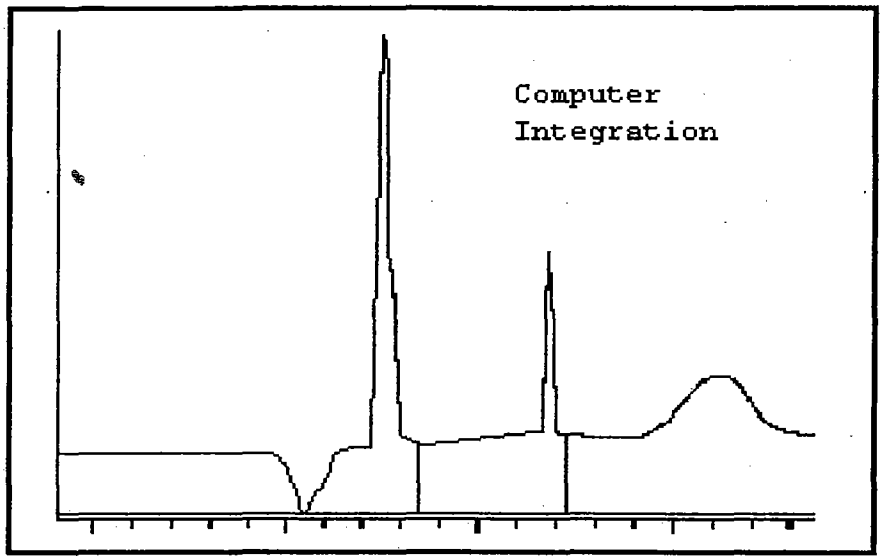


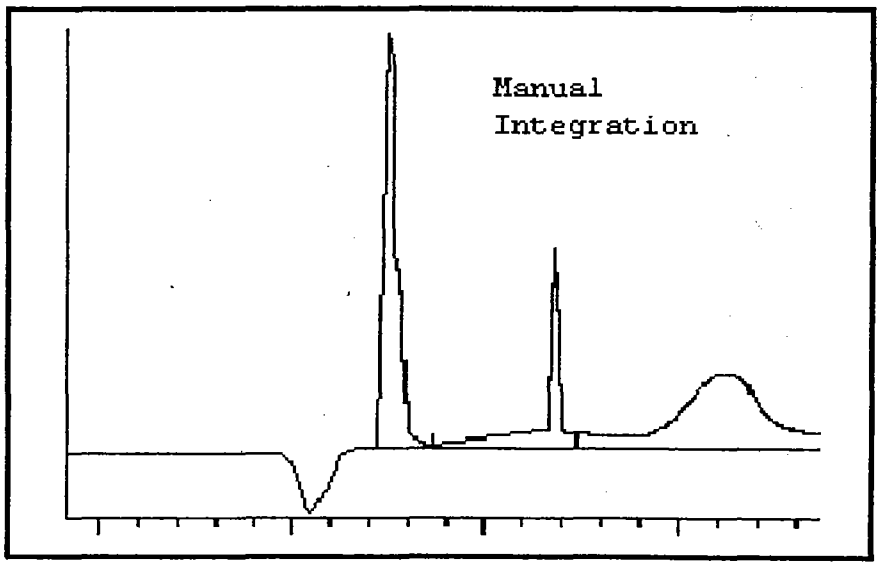


Allowable Integration

Secondary or Qualifier Ions

Attachment 5

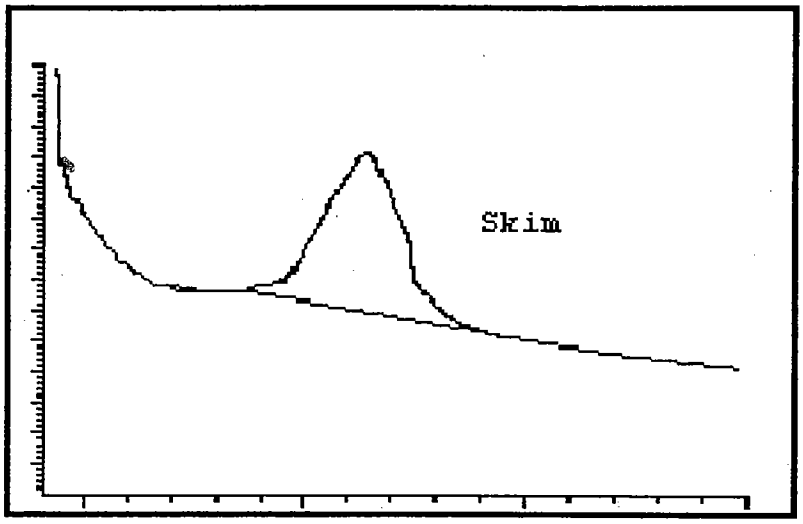


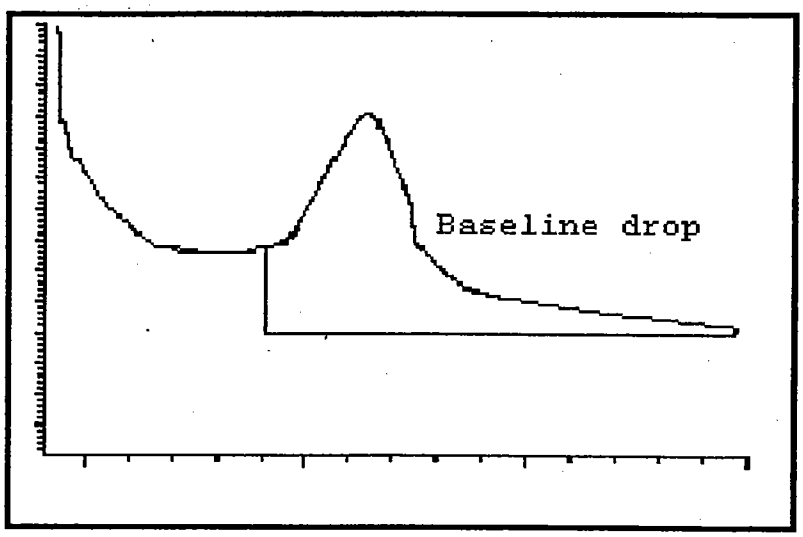


Allowable Integration

Baseline Shift

Attachment 6

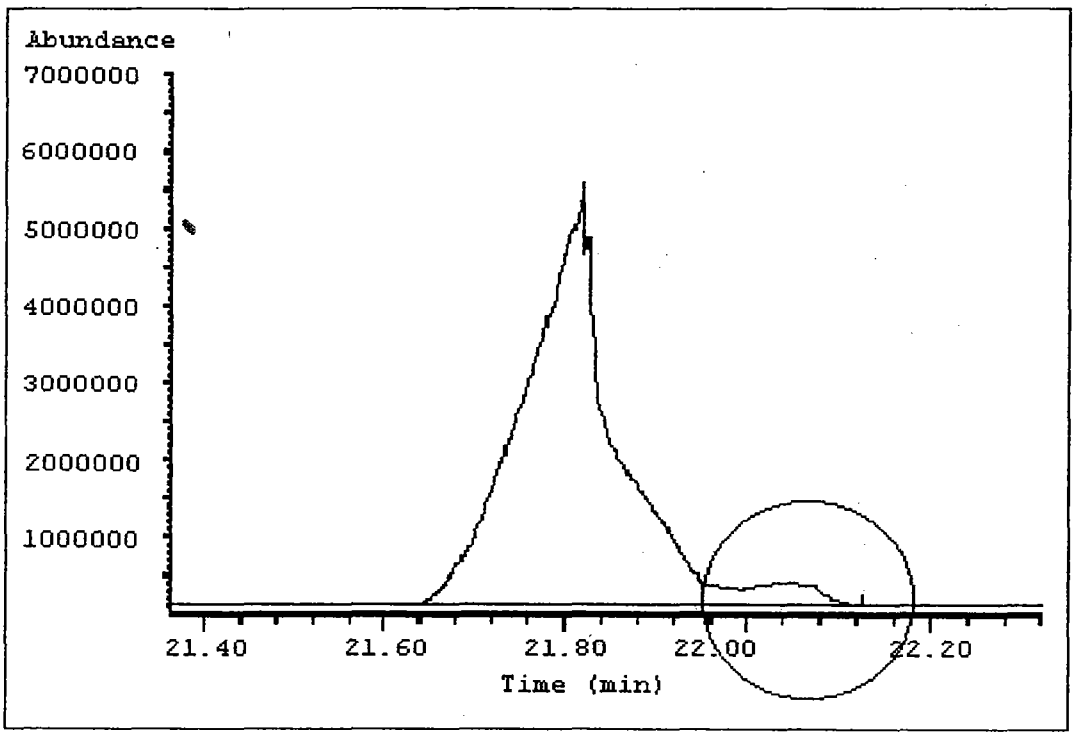




Allowable Integration

Skimming (manual) vs. Baseline Drop (computer)

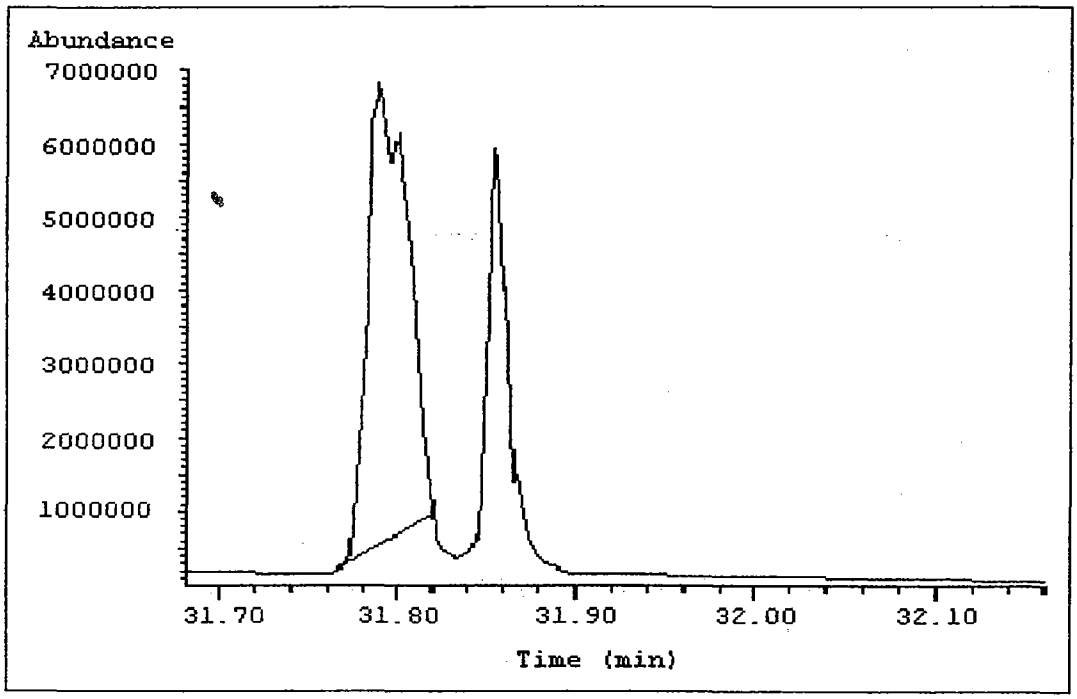
Attachment 7



Improper Integration

Adding Area by Including Other Peaks

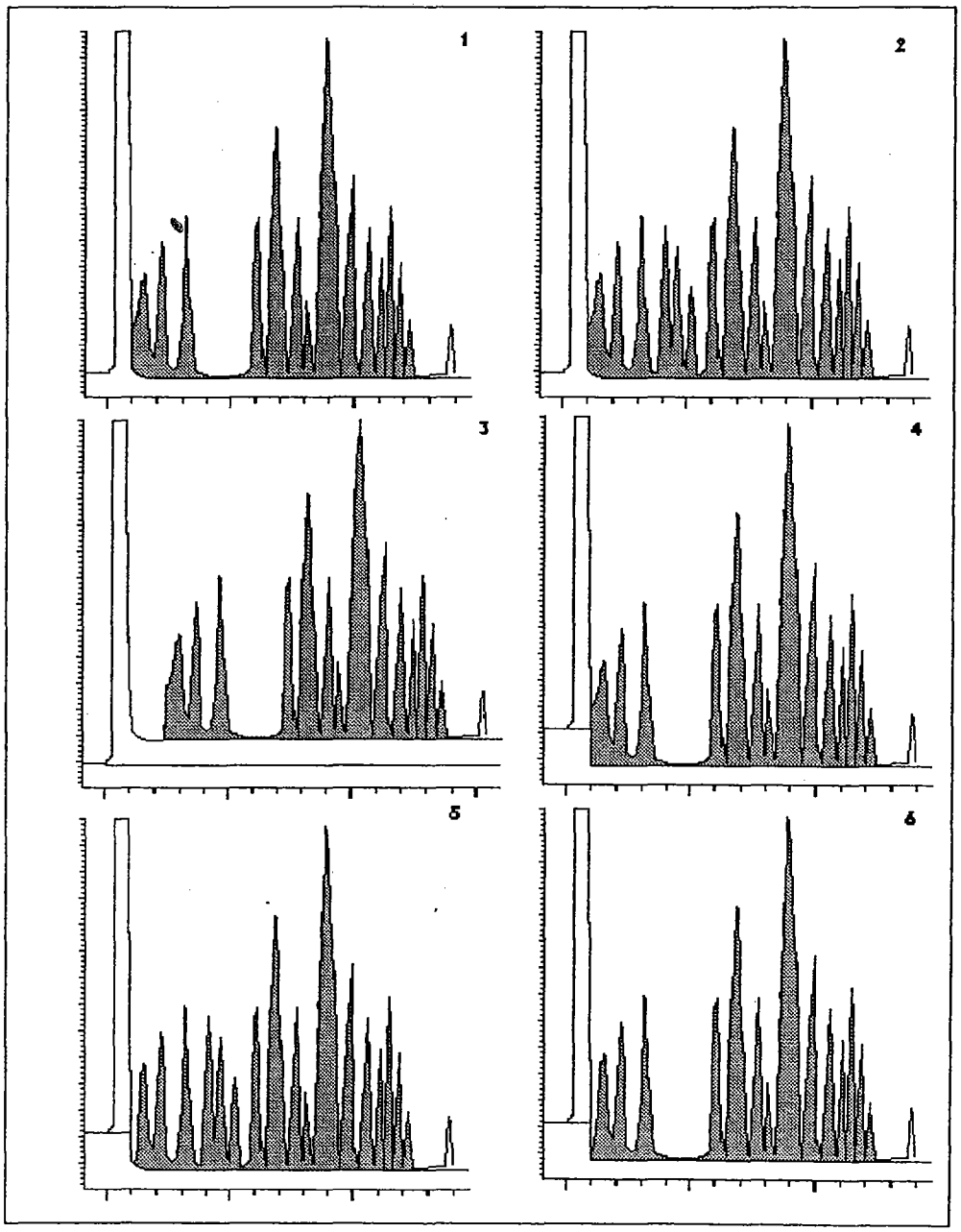
Attachment 8



Improper Integration

Baseline Moved To Decrease Peak Area

Attachment 9



Acceptable Methods of Integration
Grouped Peaks

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-2001

Title: **Waste Segregation and Disposal**

Revision history:

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

Written by: Harry Romberg Date: 12/15/05
Harry Romberg, Quality Assurance Officer

Approved by: Kathy Kreps Date: 1/16/05
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 Assigned to: Tetra

Table of Contents

1. INTRODUCTION AND SCOPE **3**
1.1 METHOD DESCRIPTION 3
2. EQUIPMENT LIST **3**
2.1 EQUIPMENT 3
3. SAFETY PRECAUTIONS **3**
3.1 SAFETY PRECAUTIONS 3
4. OPERATION PROCEDURES **4**
4.1 OPERATIONS APPROPRIATE TO ALL COLLECTION AREAS 4
4.2 MIXED SOLVENT WASTE 6
4.3 CHEMICAL OXYGEN DEMAND (COD) WASTE 7
4.4 SOIL SAMPLE DISPOSAL 8
4.5 ACIDIC WASTE 9
4.6 FLUORESCENT LIGHTS 10
4.7 PCB OIL WASTE DISPOSAL 10
5. REPORTS **11**
5.1 DISPOSAL PAPERWORK 11
5.2 ANNUAL REPORTING REQUIREMENTS 11
APPENDIX I **13**
HAZARDOUS WASTE STICKER 13
APPENDIX II **14**
EXAMPLE HAZARD CLASS STICKERS 14
APPENDIX III **16**
A QUICK PRIMER ON HAZARDOUS WASTE MANAGEMENT 16

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.

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

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

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. In the 940 facility, they are designated 30 gallon drums in the 940 solvent locker

4.2.2 Small, 5 gallon or less, containers of other mixed solvent waste may be collected as satellite accumulation units in the inorganics or organics instrument preparation areas but these **must** be transported to the primary drums when full. Likewise, waste solvent bottles are kept in some hoods in the 921 Extractions facility. Satellite accumulation containers must be kept closed when not in use and must be marked with the words "Hazardous Waste" or with other words that identify the contents of the container. This will most conveniently be done by using a blank Hazardous Waste Sticker. Open solvent containers are not allowed except when actually in use.

4.2.3 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 drums are stored in rooms with bermed doorways so this is not a problem. Satellite containers, however, will usually need separate containment.

4.2.4 When new materials are collected in the primary drum, a Hazardous Waste sticker should be affixed with an initial collection date. The Hazardous Waste stickers should be marked with a DOT shipping name of **"Waste Flammable Liquids"**.

4.2.5 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).

4.2.6 About 2 inches of headspace must be left between the top of the liquid and the top of the drum to allow for expansion.

4.2.7 When 3 or more full 55 gallon drums of this waste have been accumulated at 921 (or two drums and a drum of soil), or 1 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.

4.2.8 At the time of this writing, Laucks uses Univar as the facility of choice for handling this waste stream, although this could be changed at the discretion of QA on either a one-time or ongoing basis.

4.3 Chemical Oxygen Demand (COD) Waste

4.3.1 The primary constituents of this waste are sulfuric acid, water, mercury, silver, and chromium (both tri- and hexavalent). The collection point for this waste is in the inorganics area where CODs are analyzed. These analyses are conducted in small pre-packaged tubes. The reacted tubes are not considered to be waste until they are poured out of the tubes into a collection container.

4.3.2 Collection containers must be labeled with a Hazardous Waste sticker as previously noted. The Hazardous Waste Sticker should be marked with a DOT shipping name of **"Waste Corrosive Liquids, Acidic, Inorganic"**.

4.3.3 In addition to the hazardous waste sticker, these containers should be labeled with a corrosive (8) sticker (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.**

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 Univar 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. When samples are signed-out from their storage areas for disposal, the log sheet must be marked with the appropriate assigned drum number. This will enable the laboratory to track which samples were disposed in which drum. The tracking mechanism may change as the laboratory implements bar coding of both samples and storage locations, which will include the final waste disposal drum into which soils are dumped. That mechanism will be more fully described during the next review cycle of this SOP after the tracking system has been fully implemented.

4.4.3.1 The drums should be marked with the year the accumulation is STARTED, location from which they originate, and sequential number. Thus drums for which accumulation began in 2004 from the extractions laboratory would be marked 04-921-01. The -01 being a sequential number that would be incremented with each additional drum -02, -03, etc. throughout 2004. A drum from the main lab would be designated 04-940-01, etc.

4.4.3.2 Laucks frequently receives soil samples from outside the continental U.S. This requires a USDA APHIS soil import permit. 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.

4.4.3.3 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.). Those with >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 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.

4.4.3.4 When samples are transferred from the storage locations to the drums, the Secure Storage Custody Log must be marked to indicate into which drum they were disposed. This should include any bottle identifiers, if necessary to identify just what was disposed. Thus, it will be necessary for personnel disposing of samples to check the drums to make sure there is enough room for the designated samples. Soil samples will generally have their lids removed and disposed in the regular garbage. If the contents of the jar are easily poured into the drum, this is done and the jar disposed in the waste glass. If not, jar and all will then be disposed in the waste drum. If the lids themselves contain client identifying marks or locations or have significant amounts of adhering material (oil, etc.) which cannot be readily dumped into the drum the lid will also be disposed into the waste drum.

4.4.3.5 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 Univar 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.

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 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. In 2004, Laucks submitted these through the online version of

"Turbowaste" directly to the WDOE website. We expect to continue using the latter method of reporting. Details of this report are not provided in this SOP.

5.2.3 In addition, as part of a WDOE program to reduce hazardous waste in general, Laucks files an annual pollution prevention plan update by September 1 of each year. 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.

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._____

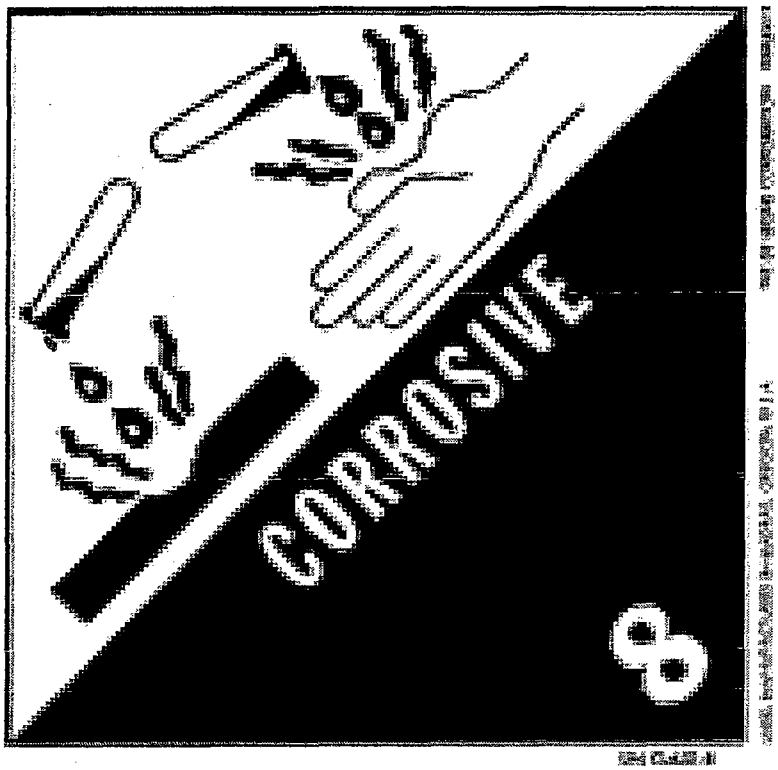
HANDLE WITH CARE!

CONTAINS HAZARDOUS OR TOXIC WASTES

STYLE WM-10

Appendix II

Example Hazard Class Stickers


CORROSIVE
8

9
Chicago, IL 60646 (800) 621-5808 HMIS1
Printed by Labelmaster, An American Labelmark Co.

Appendix III

A Quick Primer on Hazardous Waste Management

A Quick Primer on Hazardous Waste Management

Laucks is concerned and is indeed obligated by Washington State, to properly manage hazardous waste generated by the laboratory. This document is a quick overview for staff outlining the processes of which individuals should be aware. Everything is outlined in greater detail in Laucks SOP LTL-2001 *Waste segregation and Disposal*.

We currently manage 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 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 transferred to the acid storage locker. A new 5 gallon container should be started and QA should be contacted for the required labeling. Waste is not to be dumped into any carboy until it has been properly labeled.

- Soil – Waste soil samples are assumed to contain hazardous metals, specifically lead. Unless a soil is known to have been an agricultural soil or is known not to contain hazardous substances, it is treated as though it is hazardous. The soil samples are dumped into a 55 gallon drum in the 940 warehouse. When the drum is full, it is sealed, the current date marked under the "accumulation start date" on the Hazardous Waste label, and QA notified in order to arrange transport to a disposal facility. A new drum will be labeled by QA. Soil is not to be dumped into any drum until it has been properly labeled.

- Extract Vials – Extract vials are collected in the covered, labeled 5 gallon bucket in the organic standards hood. When the bucket is full to the point that it can just be closed and sealed, it should be. The current date should then be marked 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, the current date marked under the "accumulation start date" on the Hazardous Waste label, and QA notified in order to arrange transport to a disposal facility. A new drum will be labeled by QA. Soil is not to be dumped into any drum until it has been properly labeled.

- Fluorescent Lights – Fluorescent lights are considered "Universal Waste" and must never be disposed in the garbage. They must be collected and shipped to a recycling or hazardous waste disposal facility. These are collected in the boxes in which the replacement bulbs come, located in the 940 warehouse.

- PCB 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.

Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Water (8081B/8082A by 3510C)

Method # LTL-3202

April 11, 2005

Revision Number: #6

Written by: B. O'Neill Date: 4-11-05

Reviewed by: Harry Pombrey Date: 4-12-05

Approved by: Kathy Grupp Date: 4-21-05

Controlled Document

No. 20 Assigned to: Tetra

1. **PURPOSE** - In this method, organochlorine pesticides and polychlorinated biphenyls are extracted from neutral water with methylene chloride. The extracts are dehydrated, concentrated in a Kuderna-Danish apparatus and exchanged into hexane. If necessary, the pesticide/PCB extracts may be SPE Florisil cleaned prior to GC analysis. PCB only extracts may be acid cleaned prior to GC analysis.

2. **SAFETY** - During the conduct of this method, the analyst will be exposed to a variety of reagent chemicals and solvents. The health effects of these various chemicals may be ascertained by reading the material safety data sheets (MSDS) available in the general files. Additionally, the samples by their very nature, may contain significant levels of hazardous materials. It is incumbent on each extractionist to exercise due care and caution while executing this method. The company will provide any protective equipment or clothing needed to assure employee safety.

3. **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. 10 N Sodium Hydroxide - weigh 400 grams sodium hydroxide, dissolve and dilute to one liter with DIW.

4.3.2. 1:1 Sulfuric Acid - measure equal volumes of concentrated sulfuric acid and DIW. Note: Observe proper acid preparation techniques.

4.3.3. Anhydrous Sodium Sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

4.3.4. Surrogate solution prepared in acetone:

2, 4, 5, 6-Tetrachloro-m-xylene	1.0 µg/mL
Decachlorobiphenyl	1.0 µg/mL

4.3.5. 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
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

4.3.6. PCB (AR1660) matrix spiking solution prepared in acetone:

Aroclor 1016	25 µg/mL
Aroclor 1260	25 µg/mL

5. **EQUIPMENT**

5.1. Glas-Col mechanical separatory funnel shaker

5.2. Organomations Assoc., Inc. - N-EVAP, (Nitrogen Evaporator), Model 112

5.3. Vortex mixer

5.4. Standard laboratory glassware to include:

5.4.1. 2000 mL Teflon separatory funnels

5.4.2. 300 mL Fleakers

5.4.3. K-D apparatus: 250 mL. K-D flask, 10 mL or 25 mL ampule and three-ball snyder column.

5.5. All glassware 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.

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. Water samples must be extracted within 7 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 demonstarated the ability to perform the described extraction procedures and techniques.

9. **QUALITY CONTROL** - The normal level of quality control will consist of blank, blank spikes matrix spike and matrix spike duplicate (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

10. **METHODOLOGY**

10.1. Sample Extraction

10.1.1. Mix sample thoroughly in its original container and place labeling tape at sample level. Mark meniscus of sample on the tape.

10.1.2. Transfer entire contents of container into a 2000 mL separatory funnel. Rinse container with methylene chloride and add to funnel. Fill sample container to the mark on tape with water. Measure volume of sample used in a calibrated 1000 mL cylinder.

10.1.3. Prepare a blank and a blank spike with 1000 mL DIW. Prepare separate blank spike for Pesticides and PCB.

10.1.4. 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.

10.1.5. Pipet 200 ul surrogate solution into each funnel.

10.1.6. Pipet 200 ul matrix spiking solution into each QC funnel.

10.1.7. Adjust pH to 7 ± 2.

10.1.8. Add 60 mL methylene chloride to the separatory funnel.

10.1.9. Shake the samples hard for three minutes on the mechanical shaker, with the stopcocks closed.

10.1.10. Replace the funnels in rack and allow to stand for 10 minutes.

10.1.11. Drain and collect the lower methylene chloride layer in a fleaker.

10.1.12. Repeat from 10.1.8. until a total of three extractions have been performed.

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 10.1.12. 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 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 K-D apparatus into a hot water bath, using a bath temperature of 90°C, with a long ampule immersed to a depth of 8 mL. Regulate the evaporation time to take approx. 45 minutes.

10.3.3. Reduce the volume to 4-5 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 4-5 mL.

10.3.3.2. Repeat 10.3.3.1. two additional times.

10.3.4. Remove the apparatus from the water bath and cool to room temperature.

10.3.5. Rinse joint 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. Reduce the extract volume to approx. 2 mL on a nitrogen blowdown to insure removal of methylene chloride. 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.

NOTE: If necessary, the pesticide/PCB extracts may be SPE Florisil cleaned (3620B) prior to GC analysis (see LTL-3221). PCB only extracts may be sulfuric acid cleaned (3665A) prior to GC analysis (see LTL-3303).

10.3.8. Label the extract and deliver to 940.

10.3.9. Complete all necessary paperwork and bench sheet. Bench sheet to include cleanup method #, if applicable, extract location, time and date of transfer to 940. Clip the T-card on the folder and place in GC room extraction folder box. The file folder color will be purple and the blank name will be ____GPXWL_. PCB only will be a orange folder and the blank name will be ___GPB.WL_.

11. **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), 3510C (1996).

Extraction Method for Organochlorine Pesticides and Polychlorinated Biphenyls in Soil (8081A/8082 by 3550B)

Method # LTL-3302

November 29, 1999

Revision Number: #2

Written by: _____ Date: 11.29.99

Reviewed by: Harry Rowley Date: 12-13-99

Approved by: Kathy Oreps Date: 12-13-99

Controlled Document

No. 20 Assigned to: Tetra

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. The extract is split prior to any cleanup step. The extracts are GPC and SPF florisil cleaned prior to analysis by GC. The PCBs receive an additional sulfuric acid cleanup.

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. **REAGENTS**

3.1. All reagents are to be AR grade or better.

3.2. All solvents are to be distilled in glass, unless otherwise noted.

3.3. The following special reagents should be prepared:

3.3.1. 1:1 methylene chloride/acetone (v/v)- prepared by mixing equal volumes of the solvents.

3.3.2. 9:1 Hexane/acetone - prepared by adding 10.0 ml acetone to 90.0 ml hexane.

3.3.3. Anhydrous sodium sulfate - prepared by muffling AR grade sodium sulfate for four hours at 400°C.

3.3.4. Florisil SPE column check solution prepared in acetone:

2,4,5-Trichlorophenol	0.100 ug/ml
Gamma BHC	0.020 ug/ml
Heptachlor	0.020 ug/ml
Endrin	0.040 ug/ml
4,4-DDD	0.040 ug/ml
4,4-DDT	0.040 ug/ml
Methoxychlor	0.200 ug/ml
Alpha BHC	0.020 ug/ml
Dieldrin	0.040 ug/ml
Alpha Endosulfan	0.020 ug/ml
Tetrachloro-m-xylene	0.020 ug/ml
Decachlorobiphenyl	0.040 ug/ml

3.3.5. Surrogate solution prepared in acetone:

2,4,5,6-Tetrachloro-m-xylene	1.0 ug/ml
Decachlorobiphenyl	1.0 ug/ml

3.3.6. Pesticide/PCB matrix spiking solution prepared in acetone:

Gamma-BHC	2.5 ug/ml
Heptachlor	2.5 ug/ml
Aldrin	2.5 ug/ml
Arochlor 1260	25.0 ug/ml

4. EQUIPMENT

4.1. Heat Systems Ultrasonic Processer - Model XL2020, 550 watts - Maintain per manufacturer's instructions.

4.1.1. 3/4 inch titanium horn (#208)

4.2. Analytical Biochemical Laboratories, Inc. (ABC) Model 1002B or Model 1000 Gel-Permeation Chromatography (GPC)

4.3. Organomations Assoc., Inc. - N-EVAP (nitrogen evaporator), Model 112

4.4. Analytechem International - Vac Elaut SPS24 (for SPE florisil columns)

4.5. Florisil SPE cartridges with Teflon frits (1000 mg)

4.6. Standard laboratory glassware to include:

4.6.1. 8 ounce extraction bottle

4.6.2. 500 ml Fleakers

4.6.3. K-D apparatus: 500 ml. K-D flask, 10 ml or 25 ml ampule and three-ball snyder column.

4.7. All glassware to be rinsed as follows, prior to use:

4.7.1. Technical grade acetone (if the glassware is wet).

4.7.2. Triple rinsed with methylene chloride.

4.8. Volumetric measurements are to be made with a calibrated fixed or adjustable volume microdispenser and individually calibrated vials.

5. **QUALITY CONTROL** - The normal level of quality control will consist of blanks, blank spikes, matrix spikes and matrix spike duplicates (MS and MSD). This is performed on a per batch basis to include no more than 20 samples. The level of quality control will be indicated to the extractionist at the time the job is assigned. These samples serve to provide a measure of the recovery efficiency for the analyte and to provide data for statistical evaluation of the sample. In those instances that a client requires additional or different quality control measures, the extractionist will be directed accordingly in writing.

6. **METHODOLOGY**

6.1. Sample Extraction

6.1.1. Mix sample thoroughly in its original container if there is space available (otherwise mix in solvent rinsed aluminum tray).

6.1.2. Weigh 30.0 grams of soil (wet weight) into a extraction bottle.

6.1.3. Prepare two additional aliquots of a sample if Quality Control is required.

6.1.4. Add 60 grams sodium sulfate and mix well to give the soil a sandy texture.

6.1.5. Prepare a blank and a blank spike with 60 grams sodium sulfate.

6.1.6. Pipet 200 ul surrogate solution to each bottle.

6.1.7. Pipet 200 ul matrix spiking solution to each of the QC bottles.

6.1.8. Add 100 ml 1:1 methylene chloride/acetone to each bottle.

6.1.9. Sonicate the bottles for 3 minutes using the sonic horn, set at 50% duty cycle and full output (10).

6.1.10. Centrifuge the samples for 20 minutes at 2000 rpm if necessary to achieve a partition.

6.1.11. Decant off and collect the supernatant.

6.1.12. Repeat from steps 6.1.8. two additional times, combining all of the extracts.

6.2. Solvent dehydration

6.2.1. Prepare a glass funnel by plugging with glass wool, and filling 1/2-2/3 full with sodium sulfate.

6.2.2. Pre-rinse the sodium sulfate by passing 40 ml methylene chloride through the prepared funnel.

6.2.3. Pass the extract from step 6.1.12. through the funnel and collect in an assembled K-D apparatus.

6.2.4. Rinse the collection vessel with several 10 ml methylene chloride rinses.

6.2.5. Rinse the sodium sulfate with 40 ml of methylene chloride.

6.3. Solvent evaporation

6.3.1. Assemble the full K-D apparatus with a snyder column prewet with 2-3 ml of methylene chloride.

6.3.2. Immerse K-D apparatus into a hot water bath, using a bath temperature of 90° C, with a long ampule immersed to a depth of 12 ml. Regulate the evaporation time to take one to one and a half hours.

6.3.3. Reduce the volume to 4-5 ml, remove the apparatus from the water bath and cool to room temperature.

6.3.4. Rinse joint and remove snyder column. Allow rinse solvent to drain into ampule.

6.3.5. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of solvent.

6.3.6. 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.

NOTE: In instances where sample extracts are being prepared for PCB only (8082), and the GPC step is being omitted prior to acid cleanup, the solvent exchange step (see 6.3.8.1.) 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 6.4.

6.3.7. At this point the extract will be GPC cleaned -- see Method # LTL-3692.

6.3.8. K-D the GPC cleaned extract to 4-5 ml and exchange into hexane as follows:

6.3.8.1. 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.

6.3.8.2. Repeat 6.3.8.1. two additional times.

6.3.9. Remove the apparatus from the water bath and cool to room temperature.

6.3.10. Rinse joint with hexane and remove snyder column. Allow rinse solvent to drain into ampule.

6.3.11. Remove the ampule clamp, and wipe the joint with a Kimwipe. Separate the ampule and rinse the joint with a small amount of hexane.

6.3.12. Reduce the extract to less than four ml in a nitrogen blowdown. Transfer extract to a 16 x 100 mm culture tube and adjust to a final volume of 5.0 ml (as compared to a measured volume) in hexane.

6.4. Sulfuric Acid Cleanup (PCBs only)

6.4.1. Transfer a two ml aliquot from the 5.0 ml final volume (step 6.3.12.) to a 16 x 100 mm culture tube.

6.4.2. Add 2.0 mls concentrated sulfuric acid and vortex for 30 - 60 seconds.

6.4.3. Let stand for a few minutes to allow layers to separate. May be centrifuged.

6.5. Florisil Cleanup

6.5.1. Attach the vacuum manifold to the vacuum pump with a trap in between. Place a labeled 16 x 100 mm culture tube in the proper collection slot. Secure the manifold with the straps and move the manifold to the waste position. Adjust the vacuum pressure in the manifold to between five and ten pounds of pressure.

6.5.2. Place one florisil cartridge into the vacuum manifold.

6.5.3. Start the vacuum and prewet the cartridge with hexane/acetone (9:1), by passing at least 5 ml through the cartridge. Do not allow the cartridge to go dry after wetting.

6.5.4. Release the vacuum, and move the manifold to the collect position.

6.5.5. Add 1.0 ml extract from step 6.3.12. (Pesticides) or from step 6.4.3. (PCBs). to the top frit of the florisil cartridge. (Store remaining 4.0/3.0 ml of extract.)

6.5.6. Restore the vacuum, and elute the column with 8-9 ml of hexane/acetone (9:1). Allow the cartridge to go dry.

6.5.7. Release the vacuum, and move the manifold to the waste position. Remove and discard the used florisil cartridge.

6.5.8. Repeat from step 6.5.2. for additional extracts.

6.5.9. Reduce the extract volume to 1.0 ml in a warm water bath with nitrogen. Rinse internal walls of culture tube several times during blowdown.

6.5.10. Transfer extract to a 1.8 ml vial. The volume is then adjusted to 1.0 ml (as compared to a measured volume) in hexane.

6.5.11. Label the extract and deliver to 940.

6.5.12. Complete all necessary paperwork and bench sheet. Bench sheet to include the date of GPC, florisil lot number, date and time of transfer to 940 and extract location. Clip the T-card on the folder and place in the GC room extraction folder box. The file folder color will be purple and the blank name will be ____GPX.SL_.

7. **ANALYSIS TIME** - Based on experience in the laboratory, it is anticipated that a single sample may be completed in about 12 hours. If it is possible to batch similar samples it is expected that about seven samples could be completed in approximately 20 working hours. About four and a half hours of hands on time will be required of the extractionist. These approximations are based on the assumption that the samples are "average", and will not require additional time beyond normal operations. Additional time must be allocated for samples which are very dirty or are extraordinary, such as tissues or vegetable matter.

8. **REFERENCES** - The following USEPA methods are the official methods on which this Laucks Testing Laboratory method is based. The primary methods are those which most closely parallel the Laucks procedure and are referenced by their USEPA series and number. In those instances for which there are no official EPA methods, the most suitable reference is given under the miscellaneous references section. The additional reference section cites those methods which contain additional information. These methods will frequently be official methods, which apply in part to, or support the Laucks method.

PRIMARY REFERENCES:

Test Methods for Evaluating Solid Waste, USEPA, SW-846, (1996).

8081A, 8082, 3550B.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4002

Title: **Electronic Sample Entry and Log-In**

Revision history:

Number	Date
8	7/07/04
7	2/21/03
6	3/18/99
5	6/19/96
4	12/28/95
3	4/05/95
2	6/05/90
1	4/24/90

Written by: Pam Johnson Date: 7/8/04
Pam Johnson, Sample Control

Revised by: Kathy Kreps Date: 7/8/04
Kathy Kreps, Laboratory Director

Approved by: Harry Romberg Date: 7-8-04
Harry Romberg, QA Officer

Controlled Document

No. 20 **Assigned to:** TeTra

TABLE OF CONTENTS

1. INTRODUCTION AND SCOPE 4

1.1 INTRODUCTION 4
1.2 SCOPE 4

2. EQUIPMENT LIST 4

3. SAFETY PRECAUTIONS 4

3.1 SAMPLE HANDLING 4

4. OPERATION PROCEDURES 5

4.1 SAMPLE RECEIPT 5
4.2 SAMPLE LOG-IN 8
4.3 SPECIAL IDENTIFICATION FOR SAMPLES RECEIVED FROM OVERSEAS 13
4.4 SPECIAL DOCUMENTATION PROCEDURES FOR CLP SAMPLES 13
4.5 ASSIGNMENT OF SDG NUMBERS 14
4.6 ASSIGNMENT OF LAB QUALITY CONTROL SAMPLES 14
4.7 COMPLETION OF SDG RECORDS 14
4.8 SAMPLE STORAGE 17
4.9 STORAGE LOCATIONS 18
4.10 DETERMINATION OF TESTS 19
4.11 ELECTRONIC TRANSMITTAL OF SAMPLE AND TEST REQUEST RECORDS 20
4.12 GENERATION OF INTERNAL CHAIN-OF-CUSTODY (COC) 21
4.13 SAMPLE BREAKAGE 21
4.14 SPECIAL CIRCUMSTANCES 21

APPENDIX 1 22

SAMPLE RECEIPT VERIFICATION STAMP 22

APPENDIX 2 23

EPA FORM DC-1 23

APPENDIX 3 25

NON-CLP SAMPLE RECEIPT LOG 25

APPENDIX 4 27

CLP SAMPLE RECEIPT LOG 27
SUPPLEMENTAL SAMPLE RECEIPT LOG 27

PAGE 3 OF 3 30

APPENDIX 5 31

SECURE STORAGE CUSTODY LOG 31
ORGANIC EXTRACTIONS CUSTODY LOG 31

APPENDIX 6 34

Controlled Document
Assigned to: _____ No. _____

LAUCKS TESTING LAB pH LOG FORM 34
APPENDIX 7 ..... **36**
LAUCKS TESTING LAB SAMPLE SPLIT SHEET 36
APPENDIX 8 ..... **38**
REQUIRED CONTAINERS/VOLUMES/PRESERVATION/HOLDING TIMES 38
APPENDIX 9 ..... **54**
CHECKLIST FOR ANALYSES WITH SHORT HOLDING TIMES 54

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 "SAM." References made to SAM 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 marking samples with lab numbers, and in checking for consistency of all records is of utmost importance.

1.2 Scope

1.2.1 All samples received by the laboratory are logged using the following procedures.

2. EQUIPMENT LIST

Lab coat
Disposable gloves
Respirator, dust mask
3M desk cleaner, broom, dustpan, mop
Spatula
Label gun (waterproof)
PC work station linked to SAM

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, 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.

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.

4. OPERATION PROCEDURES

4.1 Sample Receipt

4.1.1 Samples may be received by client delivery, over the front counter, via 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.
- 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 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 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, 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. CLP Sample Receipt Log (Appendix 4) is for CLP log-in procedure. **NON-CLP** Sample Receipt Log (Appendix 3) is for Laucks **NON-CLP** log-in procedure. If requested a client provided receipt form may be substituted for the Laucks sample receipt log.

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 CLP Sample Receipt Log (Appendix 4); 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.

4.1.8 For any program (such as EPA, AFCEE, NFESC, or Army Corps) or other project-related 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. The temperatures are recorded on the Supplemental Sample Receipt Log (Appendix 4). If any samples exceed the range of 4°C ± 2°C, the client must 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 Supplemental Sample Receipt Log must be kept with the COC in the workorder 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
- 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.

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 CLP Sample Receipt Log Form (Appendix 4).

4.1.16 All preserved water sample bottles for project-related work as well as unpreserved water sample bottles for AFCEE, NFESC, or Army Corps projects must also 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. 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 4). If any samples exceed the pH requirements, the client must be contacted. In most cases, 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 6) for corrective action. After the corrected preservation is completed this form is given to the appropriate project manager for workorder 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 7). There is a specific cart located in sample entry where the samples are temporarily stored until splitting and preservation takes place.

4.1.18 All sample container marks (including ID's, dates and times) are then verified with each other and with the COC. This is 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 discrepancies are noted on the Non-CLP Sample Receipt Log (Appendix 3) or the CLP Sample Receipt Log (Appendix 4) 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 8, "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 "CLP Sample Receipt Log" (see Appendix 4). 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.

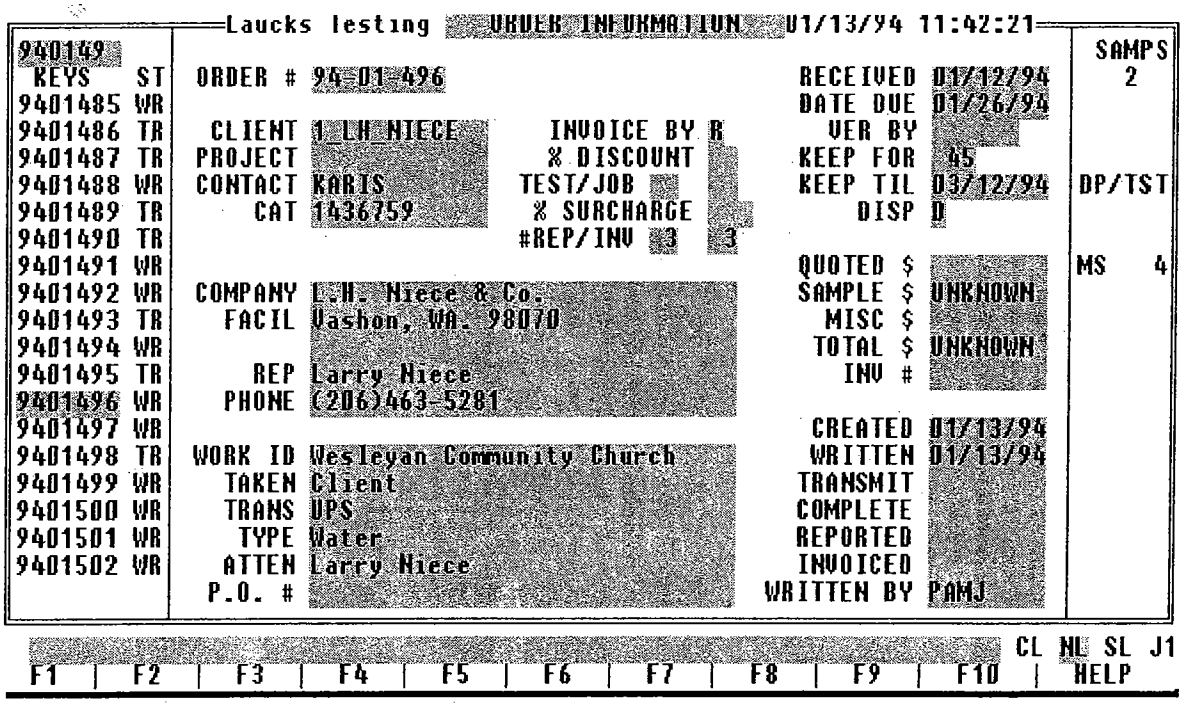
4.2 Sample Log-In

4.2.1 Determine whether a client record exists in the SAM 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 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 the client code would be project specific. Example (client name_project name).

4.2.2 A SAM workorder is started for the job through the ORD program. The workorder is identified by a unique 7-digit number, which is assigned by SAM at the time the workorder is initiated. (The first two digits of this number represent the year, the third and fourth digits represent the month, and the final three digits represent the workorder's sequence within the month. For instance, workorder 0204001 was initiated in April, 2002 and was the first workorder for that month.) This number will be used throughout the laboratory to track the job.



ORD Screen

4.2.3 The workorder is to be filled out as completely as possible at this time. Above is an example of what the workorder screen looks like on your PC. Typical information put into the workorder screen (analogous to a cover page) includes:

- date of sample receipt;
- workorder due date;
- client point of contact (if different than in the client record);
- sample type (soil, water, etc.);
- the manner in which the samples were received at the laboratory (hand-delivered, Greyhound, etc.);
- air bill number (or equivalent) if the sample was transported by common carrier;
- the client's overall project identification (both the name of the project and any project, job, or purchase order number);
- and any relevant surcharges or discounts to be applied at the time of invoice.

4.2.4 All required data entry fields are in inverse video (highlighted) on the screen, but fields for purchase order numbers, project name or number, and point of contact should also be completed, if the information is known.

4.2.5 All sample IDs, dates of collection, and dates of receipt are recorded in the FRAC program for the workorder with which they are associated. If there is a discrepancy in identification between bottles of the same sample, make a note on the appropriate sample receipt log or for EPA the DC-1 Form and the project manager will notify the client.

```
94041                Laucks Testing  FRACTION DESCRIPTION 04/05/94 13:36:36
   KEYS                 ORDER# 94-04-191/WR CON MARKB                DEPTS/TESTS
9404176-12A             CLIENT PETER PAN    CAT                                   HIST1
9404176-13A           WRITTEN 04/05/94       BY HOLLW                             HIST2
9404176-13B
9404176-13C                DASH # 02 A CATEGORY
9404176-13D           SAMPLE ID Mahi Mahi Lot #X3159 (#2)
9404176-14A           COLLECTED 04/05/94            QA/QC   MAT F
9404176-15A              STORED Analyst/Freezer   RECVD 04/05/94 SC    2
9404176-15B                                         DUE 04/15/94
9404176-15C           JOB HIST2  FL  QC?          QUOTE    75.00
9404176-15D          NAME Histamine                SDG#          StID#
9404176-16A          TESTS:DEP/CAT/NAME/D/R
9404176-17A             HISTAM
9404176-17B
9404176-17C
9404176-17D
9404176-18A          -SAMPLE AND WORK DESCRIPTION-
9404191-01A           This job code is for any additional samples.
9404191-02A
                                                                          CL NL SL J1
F5=Result,Price  F6=Lookup,NextJob,StateID   F10=More   Shft-Fn=Fn Help
```

FRAC Screen

4.2.6 All sample bottles are numbered with the workorder number and a fraction (or sample) number. Fraction numbers are assigned sequentially to each sample based on the order in which the samples were sorted and logged (COC order, client sample number order, etc.). This number is used to track the sample throughout the laboratory. See section 4.2.10 for specifications for unique bottle identifiers.

4.2.7 A workorder number and the fraction number can uniquely identify a sample. For instance, if the workorder number was 0204001 and there were 4 samples, the job would consist of samples,

0204001-01
0204001-02
0204001-03
0204001-04

4.2.8 Each sample or set of samples is assigned a unique identifying workorder number, generated by the Laboratory Information Management System (LIMS), on receipt. This unique number consists of 7 numerical characters, such as 0207215. In this example, the workorder was initiated in 2002 (02), in the month of July (07) and was the 215th such workorder that month (215). If more than 999 workorders are generated in any month, the 5th digit is replaced by successive letters of the alphabet (A-Z).

4.2.9 If necessary, more than one fraction may be created for a sample (generally, this is related to billing issues - when one analysis is discounted in price and another is not, for instance), but they will all bear the same fraction number and be differentiated by an automatically-assigned letter suffix. For instance, if sample 0204001-01 had 3 fractions, purely for internal accounting reasons, the three fractions would be identified as:

0204001-01A
0204001-01B
0204001-01C

4.2.10 The person performing log-in needs to be aware of this effect, but it has no impact on sample identification within the lab, on sample tracking, or on the sample number placed on the bottles/jars. In the above example, all bottles submitted for this sample would be marked 0204001-01.

4.2.11 Every bottle must have a specific 1-3 digit numerical identifier that is unique to each bottle submitted within a workorder. The numbers are assigned in consecutive order so that all bottles of similar size/type with the same preservation for the same analysis (analyses) from a particular workorder will have consecutive bottle numbers. The first

bottle of each analysis type in each new workorder starts over again with bottles number 1.

4.2.12 This information must be recorded in the "Bottles" computer-tracking program. Under each workorder and the workorder-unique bottle identifier will be printed in the bottom left corner of each bottle label (which also contains the workorder number and the sample number) before the bottle label is affixed to each individual bottle.

4.2.13 **ALERT:** Each SAM workorder can accommodate up to 57 fractions and no more. Each workorder must allow sufficient fraction space for later changes or additions. Therefore, no more than 50 samples should be logged into any single workorder. If, for administrative reasons, some or all of the samples consist of more than one fraction, then no more than 50 fractions can be logged. Should the submittal consist of more than 50 samples, or more than 50 fractions, initiate additional workorders as required. Cross-reference the workorder numbers, so that all samples submitted together can be reported together to the client. You can perform this cross-reference manually (by noting on accompanying documents "See Also [Workorder Number]) or you can make appropriate comments in the Workorder Comment field (F2). To the degree possible, make sure that multiple workorders which represent one complete project in the client's mind are created sequentially, with no other unrelated workorder numbers intervening.

4.2.14 Additionally, **S**ample **D**elivery **G**roups (SDGs) are commonly created for project work at the time of sample entry. SDGs consist of no more than 20 samples being analyzed for the **same** test. This is in order that the appropriate amount of QC may be analyzed and reported with any sample set. Specifics of the SDG creation process are outlined elsewhere in this SOP.

4.2.15 **ALERT:** Each fraction will accommodate only 27 tests. If more than 27 analyses are required on any sample, additional fractions should be made (i.e. -1A, -1B, -1C, etc.).

4.2.16 **ALERT:** The workorder will accommodate only one date of receipt, while each fraction will accommodate individual receipt dates and due dates. If samples are submitted over several days, and are logged into one workorder, the Sample Custodian **MUST** enter appropriate dates of receipt in each fraction. The FRAC program will default to the current date. If the samples were received on an earlier date, that date **MUST** be entered for that fraction for the date to be correct. Similarly, the fraction due date will default to that of the workorder on the ORD screen. If different fractions of the same sample are due at different times, due to client or other demands, the date they are due **MUST** be entered for that fraction.

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, 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.

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 CLP Sample Receipt Log and the Supplemental Sample Receipt Log are CLP-specific sample login sheets. For each cooler received a CLP Sample Receipt Log Form and a Supplemental Sample Receipt Log must be completed. This form takes the place of the NON-CLP Sample Receipt Log (Appendix 3). Copies of these forms may be found in Appendix 4.

4.4.3 Complete the header information requested at the top of the forms. Use multiple pages if necessary.

- date received
- time received
- client name
- SDG #
- COC # (if available)
- sample log-in date
- workorder #
- client project
- airbill number (if available)
- and initials of the person logging in the samples.

4.4.4 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 workorder file.

4.4.5 Since the extractable fractions will be transferred to the extractions lab, a Secure Storage Custody Log must be completed, and the samples are held on 8C in the WO1 walk-in cooler (extractions hold shelf) pending pick-up by extractions personnel. Specifics of the Storage Custody Log are outlined in the Chain-Of-Custody SOP (LTL-1003) located in the SOP manual.

4.5 Assignment of SDG numbers

4.5.1 The SDG name is assigned by sample control and is usually based on client name or project name followed by sequential numbering.

4.6 Assignment of lab quality control samples

4.6.1 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.

4.6.2 Note in the **SAM SDG** records which sample is QC-assigned.

4.6.2.1 This information is pulled into SDG from the FRAC program when the SDG is created.

4.6.2.2 On the FRACTION screen, an X is placed in the QC field. This will print out next to the sample and indicate that it is an assigned QC sample.

4.6.2.3 Write a department comments message for whichever department (EX, MS, and/or GC) should be notified. Department comments for extractions should be entered in the comment section of each fraction screen (as that is where they look for the comments – they don't look at department comments).

4.7 Completion of SDG records.

4.7.1 For each project (AFCEE, NFESC, or Army Corps), CLP SDG records in SAM are created. The SDG concept follows the CLP model: that is, up to 20 samples of similar matrix and analytical fraction are grouped together for preparation and analysis. Samples are assigned to SDGs at sample login and are also reported by SDG.

4.7.2 The **SDG** program is an electronic means of compiling information about the samples assigned to an SDG in one place. Example **SDG** screens are reproduced on the following page:

```
                                   SDG Database
CDM      SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G   Project   : CDM (EDB)                 Client:
BOG05I   SAS Number:          Case Number:          Max. Samps:
BOG06G
BOG06I    Work  Samp                                                Associated
BOG07G    Ord   Num  QC            Client ID          Mat TS pH      Blank
BOG07I
BRPTSS
CANONS   9302598 17     07-MW-005-0-000-F              W
CB2SFV   9302598 20     07-MW-007-0-000-F              W
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                  CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 1

```
                                   SDG Database
CDM      SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G   Project   : CDM (EDB)                 Client:
BOG05I   SAS Number:          Case Number:          Max. Samps:
BOG06G
BOG06I    Work  Samp            Date
BOG07G    Ord   Num   VTSR    Collected
BOG07I
BRPTSS
CANONS   9302598 17  02/16/93 02/13/93
CB2SFV   9302598 20  02/16/93 02/13/93
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                                  CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 2

```
                                   SDG Database
CDM      SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G   Project   : CDM (EDB)              Client:
BOG05I   SAS Number:          Case Number:          Max. Samps:
BOG06G
BOG06I    Work  Samp                   Fractions
BOG07G    Ord   Num   EDB
BOG07I
BRPTSS
CANONS   9302598 17    X
CB2SFV   9302598 20    X
CB3-3V
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                          CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 3

```
                                   SDG Database
CDM      SDG Group : CDM10        Date Due: 03/13/93        Created:  02/19/93
  KEYS   Fraction  : E SPVMITO                              Updated:  02/19/93
BOG05G   Project   : CDM (EDB)              Client:
BOG05I   SAS Number:          Case Number:          Max. Samps:
BOG06G
BOG06I
BOG07G
BOG07I
BRPTSS
CANONS
CB2SFV
CB3-3V   Comments:
CBSFXS
CDM10E
CDM10G
CDM10I
CDM10P
CDM10S
CDM10V
CDM11E
CDM11G
                                                          CL NL SL J1
5=Lookup,Copy,Print  6=Toggle     F10=More    Shft-Fn=Fn Help
```

SDG, Screen 4

4.7.3 Fill in the header section of the first screen page. For CLP cases, fill in the Fraction (V=Volatiles, P=Pesticides, S=Semivolatiles etc.) Indicate the project name, and the client's name.

4.7.4 When the workorder number and sample number are entered, the sample-specific information shown in screens 1, 2, and 3 is read in from the SAM database. (**Hint**: after the first workorder number is entered, it is only necessary to enter sample numbers for subsequent samples from the same workorder.)

4.7.5 On screen 3, a table of fractions/tests is created. An 'X' is entered to signify that a particular test is required on a given sample.

4.7.6 Each 'fraction' has a separate SDG entry. For instance, VOAs and ABNs are entered on separate SDG records, as indicated above (V=Volatiles, S=Semivolatiles, P=Pesticides). A single letter suffix (V, S, P etc...) is assigned to each SDG record before it is saved to disk. The end result is that you may have multiple SDG records for a given SDG, each with the same root name, but a different suffix. This system is used to allow for the possibility that within the same SDG, varying numbers of tests will be assigned to samples within that SDG.

4.7.7 The last screen page is used for comments, which the sample login person or project manager would like to record for the operations staff. In general, this would include analyte lists or more information on method requirements.

4.8 Sample storage

4.8.1 The following tests must be started very soon after receipt when performed on water samples.

Test Type/Name	SAM Code(s)
NO_3 – nitrate	NO3ICW
NO_2 – nitrite	NO2_W, NO2_DW
ortho phosphate and soluble reactive phosphate	P04O_W, P04S_W
Cr^{+6} - hexavalent chromium	CR6_W, CR6_WM
CO_2 - carbon dioxide	CO2_N
DO - dissolved oxygen	DO_W
BOD - Biological Oxygen Demand	BOD_5
Chlor A – Chlorophyll A	CHLORA
Settleable Solids	SETSOL, SETSL2
Filtration for dissolved metals	FILTER
PH	PH_EPW, PH_SWW
Microbiological tests	[various]

Color	COL_DW
Turbidity	TUR_TW, TUR_W
Sulfite	SO3_W
MBAS - Methylene Blue Active Substances	MBAS
Chlorine	CL2_R

4.8.2 A rush backlog report is printed throughout the day for short holding-time tests, with the exception of microbiology, in order that they be recognized by the analysts. **A checklist (Appendix 9) for analyses with short holding times is completed prior to release of samples to the laboratory. Sample management will verify that the correct RUSH test codes are entered, date, time received and collected dates are accurate and to ensure matrices and sample I.D.s are correct at time of log-in.**

4.8.3 If microbiological samples and samples that 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 entering area.

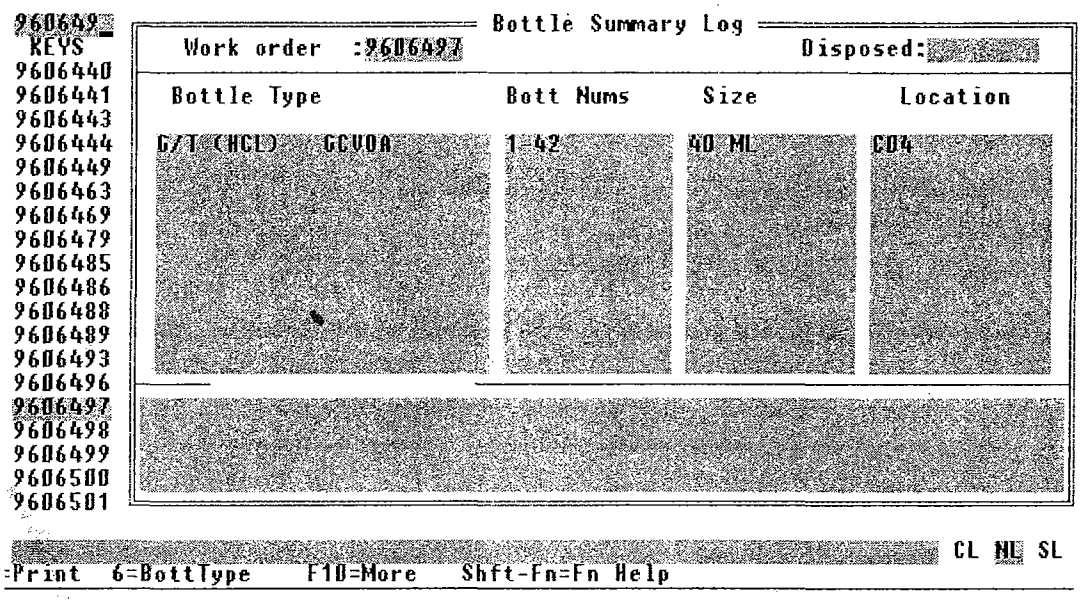
4.9 Storage locations

Location	**Description of contents**
VOA refrigerator	Soil and water volatiles
Shelf	Aqueous metals, oils, no cooling required
Inorganics cooler	Complete small water jobs
Walk-in cooler	All other soil and water*
Section in the walk-in cooler indicated w/CLP sign-in, sign-out sheets.	All samples that are under internal COC. CLP samples are also stored in here.

4.9.1 *See Section 4.7 for transfer of extractable aliquots to the extractions lab

4.9.2 Prior to putting bottles into any storage location, the electronic Bottle Summary Log must be completed. Unique bottle identifiers must also be entered in the bottle log.

4.9.3 At the J1 prompt, type "BOTTLES." This log details how many bottles were received, what type and size of bottles were received, the storage location of the bottles and the bottle numbers, where applicable. An example of this log follows.



4.10 Determination of tests

4.10.1 If Laucks provided sample bottles for the client, the bottle order, the client COC, file notes, letters, client instructions, or the client file are consulted as necessary to determine what tests are to be performed. The type of bottles received for water samples will help greatly in determining which tests to perform. If you can't determine the tests, give the paperwork to the Project Manager, who will contact the client.

4.10.2 A lab work request is initiated at this time. Based on a review of the above information, test codes are assigned to the appropriate fractions. These test codes may represent single-data point analyses ("regular" tests) or multiple-data point analyses ("special" tests), such as GC/MS volatiles. However, no work request packet can be prepared until after "transmittal," which is initiated by the Project Manager or designee.

4.10.3 Some soil samples will need to be shared between two or more departments. Place comments in department comments (or in the fraction comment section for extractions) 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:

4.10.4 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:

4.10.4.1 Notify extractions vial email that the sample is available and log 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 log it into an available shelf in the walk-in refrigerator at 940.

4.10.4.2 Notify inorganics that the sample is available and log the sample into an available shelf in the walk-in refrigerator

4.11 Electronic Transmittal of Sample and Test Request Records

4.11.1 Specifics of the transmittal process are detailed in a separate SOP. A brief summary follows. For actual transmittal, that SOP should be referenced as it will detail greater specifics and will contain changes that may occur in the transmittal process. The following is only intended as a brief overview and may not reflect the most current practices.

4.11.2 All documentation (including, but not limited to, air bills, chain-of-custody documents, bottle order forms, notes, contracts, messages, letters, etc.) that supports the information entered into the workorder and the sample fractions is clipped together by the Sample Custodian when sample log-in is complete. The workorder number is written on, at a minimum, the chain-of-custody document and may also be written on any other relevant documents.

4.11.3 The supporting documentation is given to the project manager, her designee, or to the head of the Project Management Group for "transmittal." Transmittal is the electronic approval of the workorder and sample fractions as written and must be performed within 1 working day of sample log-in. Transmittal is the activity, which electronically puts the samples and test requests into the laboratory's analytical schedule.

4.11.4 In performing the transmittal, it is the responsibility of the Project Manager, or designee, to double-check the workorder and test fractions for the following:

- accuracy of project information (number, name, point of contact, etc.)
- accuracy of test requests
- and accuracy of the test codes employed to represent those test requests.

4.11.5 The Project Manager makes corrections to these items as necessary, usually in consultation with the Sample Custodian. When transmittal is complete, the hard-copy record generated in the transmittal process is stapled to the supporting documentation previously assembled by the Sample Custodian and the complete record is filed alphabetically (by client name) in the filing drawer designated. If a CLP-style package is being generated, packets are prepared for the CLP Document Control Custodian.

4.11.6 Specific test requests are made known to analysts through hard-copy work "backlogs". For a description of this process, see the SOP on Data Handling.

4.12 Generation of internal Chain-of-Custody (COC)

4.12.1 Samples, which must be removed from the main building at 940 and taken to the extraction lab at 921 for preparation, are tracked with an internal COC. The sample custodian logging in the samples initiates this form. The lab number, the project, the number of samples, the sample matrix and the sample custodians initials and the date and time the form is started are recorded. See Appendix 5 for an example of an internal COC.

4.12.2 The samples are placed on shelf 8C in the walk-in with the COC. The person removing the samples from 940 signs and dates the form. The samples are logged into a logbook at 921 before being placed in the cooler.

4.13 Sample breakage

4.13.1 All sample breakage whether in shipping or while handling in the lab, must be reported immediately to the Project Manager.

4.13.2 If the sample was water, clean thoroughly with disposable towels. Be very careful with broken glass so as to avoid cuts.

4.13.3 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.

4.13.4 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.

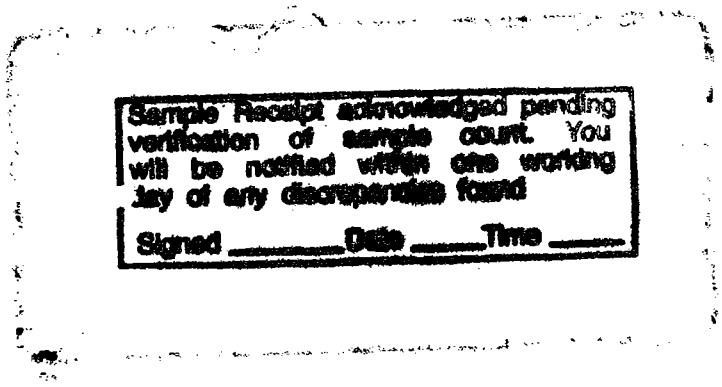
4.14 Special circumstances

4.14.1 Samples from some clients are logged into a monthly workorder. Some jobs extending over more than one sampling event may be entered under one workorder number. In that event, pay special attention to date of receipt (see ALERT, above).

4.14.2 Other special circumstances may arise. If there are any questions, check with the Project Manager first.

APPENDIX 1

Sample Receipt Verification Stamp



APPENDIX 2

EPA Form DC-1

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.

Draft SOM01.X (11/2003)

APPENDIX 3

NON-CLP Sample Receipt Log

Laucks Testing Laboratories, Inc.
NON-CLP SAMPLE RECEIPT LOG

Initial once samples are checked in_____

DATE RECEIVED:_____ SAMPLE LOG-IN DATE:_____
TIME RECEIVED:_____ WORKORDER #:_____
CLIENT NAME:_____ CLIENT PROJECT:_____
SDG #_____ AIRBILL ATTACHED?:(#)_____
COC #_____ RECEIVED BY:_____

Non-Conformance: (Check applicable item(s)) _____ Client IDs affected:

°(1) Not enough sample sent for proper analysis. #s affected:_____
j(2) Sample Bottle received broken and/or cap not intact._____
j(3) Custody seal: Absent____ Present/Intact_____ Present/Broken _____
j(4) Any temperature out of compliance:_____
j(5) Sample received outside of holding time._____
j(6) Sample not properly preserved. pH = ___. Wrong preservative used._____
j(7) Illegible sample numbers or label missing from bottles._____
j(8) Identification on bottle same as identification on paperwork: yes:_____no:_____
j(9) Incomplete instructions received with sample(s), i.e.,
j no Request for Analysis, no Chain-of-Custody._____
j(10) Samples received in improper container._____
j(11) Samples held in field before receipt by Lab. Days (specify)_____
j(12) Air Bubble(s) in ___of ____ samples for volatiles analysis._____
j(13) Other _____

CORRECTIVE ACTION: (Check applicable item(s)
Correction action taken by:

	Inititals	Date
j(1) Client informed verbally (Client Services).	_____	
j(2) Client informed by memo/letter/fax (Client Services).	_____	____
j(3) Sample processed "as received" (Sample Entry).	_____	____
j(4) Re-sampling requested of client (Client Services).	_____	____
j(5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	____
j(6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	_____	____
j(7) Other (Specify)_____	_____	____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.
Comments:

APPENDIX 4

CLP Sample Receipt Log

Supplemental Sample Receipt Log

COOLER RECEIPT FORM

WORKORDER #: _____ Contractor Cooler _____
QA Lab Cooler # _____
Number of Coolers _____

Project: _____

Date samples were received at the laboratory: ___/___/___

A. PRELIMINARY EXAMINATION PHASE: Date cooler was opened: ___/___/___

By: (print)_____(sign)_____

1. Did cooler come with a shipping slip (airbill, etc.),>>>>>>>>>>>>>>>>>>>>> YES NO
   If YES, record carrier name and airbill number: _____
2. Were custody seals on outside of cooler?>>>>>>>>>>>>>>>>>>>>>>>>>>>>> YES NO
   How many and where: _____
   Seal date: ___/___/___Seal name: _____
3. Were custody seals unbroken and intact at the date and time of arrival?>>>>>> YES NO
4. Did you screen samples for radioactivity using the Geiger Counter?>>>>>>>> YES NO
5. Were custody papers sealed in a plastic bag and taped inside to the lid?>>>>>> YES NO
6. Were custody papers filled out properly (ink, signed, etc.)?>>>>>>>>>>>>>> YES NO
7. Did you sign custody papers in the appropriate place?>>>>>>>>>>>>>>>>>>>YES NO
8. Was project identifiable from custody papers? If YES, enter project name at top of this form.
9. If required, was enough cooling material present?>>>>Type of ice: _____YES NO
10. Have designated person initial here to acknowledge receipt of cooler: ____date___/___/___

B. LOG-IN PHASE : Date samples were logged-in: ___/___/___

By (print) _____(sign)_____

11. Describe type of packing in cooler: _____
12. Were all bottles sealed in separate plastic bags?>>>>>>>>>>>>>>>>>>>>>> YES NO
13. Did all bottles arrive unbroken and were labels in good condition?>>>>>>>> YES NO

Page 1 of 3

LAUCKS TESTING LABORATORIES, INC.
COOLER RECEIPT FORM (continued)

14. Were all bottle labels complete (ID, date, time signature, preservative, etc.)? YES NO
15. Did all bottle labels agree with custody papers?>>>>>>>>>>>>>>>>>>>>>>>YES NO
16. Were correct containers used for the tests indicated?>>>>>>>>>>>>>>>>>>>YES NO
17. Were correct preservatives added to samples?>>>>>>>>>>>>>>>>>>>>>>>>>YES NO
18. Was a sufficient amount of sample sent for tests indicated?>>>>>>>>>>>>>> YES NO
19. Were bubbles absent in VOA samples: If NO, list by QA #:>>>>>>>>>>>>>> YES NO
20. Was project manager called / faxed & status discussed? >>>>>>>>>>>>>>>>> YES NO
    If YES, give details below
21. Who was called / faxed? _____
    By whom?_____(date)_____

DISCREPANCIES:

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

Page 2 of 3

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

Workorder Number:_____
Assigned SDG Number:_____

Temperature		pH of Bottle Types								

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

APPENDIX 5

Secure Storage Custody Log

Organic Extractions Custody Log

Laucks Testing Laboratories, Inc.

Secure Storage Custody Log

Project:_____ **LTL Number:**_____

Number of Containers (*optional*):_____

Storage Unit:_____ **SDG Number** (*optional*):_____

Sample Numbers	Matrix (*optional*)	Location (*shelf*)	Logged Out Date	Logged Out Time	By	Logged In Date	Logged In Time	By	Action

Samples Disposed of by _____ on _____

Laucks Testing Laboratories, Inc.

Organic Extractions Custondy Log

Samples Entered By	Time	Date	JOB #	
			Client	
Samples Moved From Bldg. 940 to 921 by:			*Matrix*	*Sample #*
			SOIL	
			WATER	
			SLUDGE	
			MISC.	
			OIL	

Comments:

APPENDIX 6

Laucks Testing Lab pH Log Form

Laucks Testing Lab pH log Form

Date_____

Analyst	WO Number	Fractions	pH before	Volume Acid Added/Type	Resulting pH

APPENDIX 7

Laucks Testing Lab Sample Split Sheet

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

APPENDIX 8

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. 49, No. 209, October, 1984)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Purgeable Halocarbons	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, no headspace	14 days, with preservation	Method 601, GC/ELCD or Method 624, GC/MS
Purgeable Aromatic Hydrocarbons	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, adjust pH to $\leq$ 2 HCl, no headspace	14 days, with preservation 7 days, if not preserved	Method 602, GC/PID or Method 624, GC/MS
Acrolein and Acrylonitrile	2 - 40 ml containers	Glass, Teflon-lined Septum, 40 ml capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.5 g ascorbic acid, no headspace As above, and pH adjusted to pH 4-5.	7 days 14 days	Method 624, GC/MS
Phenols	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C, if there is presence of residual chlorine then preserve with 0.008% $Na_2S_2O_3$ pH <2 H_2SO_4.	7 days until extraction; 40 days after extraction for analysis	Method 625, GC/MS

A. PRIORITY POLLUTANTS - Organics Analysis (Federal Register Vol. 49, No. 209, October, 1984) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Pesticides (Organochlorine Pesticides, and PCB's	1 liter	Glass, Teflon-lined Septum, 1 liter or 1 gallon capacity	Cool, 4° C	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, 4° C	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, 4° C	7 days until extraction; 40 days after extraction for analysis	Method 625, GC/MS

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Volatile Organics	2 - 40 ml containers for liquids up to 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above, Encores or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH $\leq$ 2 with HCl, no headspace Cool, 4° C if contains sodium bisulfate preservative, otherwise freeze	14 days, with preservation 7 days, if not preserved 48 hours to preservation at the lab, then 14 days to analysis (measured from collection)	Method 8015, or 8021 GC/FID or PID Direct Injection or Headspace, Method 5020 Purge-and-Trap, Method 5030 or 5035 or Method 8260, GC/MS Purge-and-Trap Method 5030 or 5035

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Acrolein, Acrylonitrile Acetonitrile	2 - 40 ml containers for liquids up to 20 grams for solids	Glass, Teflon-lined Septum, 40 ml capacity Above, Encores or Glass, 2-4 oz. capacity	Cool, 4° C, adjust pH 4-5 with HCl, no headspace Cool 4° C if contains sodium bisulfate preservative, otherwise freeze	14 days, with preservation 48 hours to preservation at the lab, then 14 days to analysis (measured from collection)	Method 8030 or 8015, GC/FID Direct Injection or Headspace, Method 5020 - Purge-and-Trap Method 5030 - Groundwater using Method 5030 only. Method 8260, GC/MS Purge-and-Trap Method 5030 or 5035
Phenols	Approximately 1 liter for liquid sample 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 Cool 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8040 GC/FID or GC/ECD or Method 8270 GC/MS
Organochlorine Pesticides and PCBs	Approximately 1 liter for liquid sample 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 Cool, 4° C	Extracted within 7 days and completely analyzed within 40 days Extracted within 14 days and completely analyzed within 40 days	Method 8081/8082 GC/ECD

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Polynuclear Aromatic Hydrocarbons (PAHs)	Approximately 1 liter for liquid sample 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 Extracted within 14 days and completely analyzed within 40 days	Method 8310 HPLC or Method 8270 GC/MS
Chlorinated Hydrocarbons	Approximately 1 liter for liquid sample 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 Extracted within 14 days and completely analyzed within 40 days	Method 8270 GC/MS
Organo-phosphorus Pesticides	Approximately 1 liter for liquid sample 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 14 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 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 Extracted within 14 days and completely analyzed within 40 days	Method 8151 Extraction and Esterification/GC-ECD

B. WASTE EVALUATION - ORGANICS ANALYSIS (SW-846, 3rd Edition) (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Volatile Organics (VOAs)	2- 40 ml containers for liquid sample up to 20 grams for solids	Glass, Teflon-lined Septum 40 ml capacity As above, Encores or glass, 2-4 oz.	Cool, 4° C, acid preserved with HCl to pH < 2, no headspace Cool, 4° C if contains sodium bisulfate preservative, otherwise freeze	14 days 48 hours to preservation at the lab, then 14 days to analysis (measured from collection)	Method 8260 Purge-and-Trap GC/MS, prep Method 5030 or 5035
Semi-Volatile Organics	Approximately 1 liter for liquid sample 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 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 Approximately 200 grams for solid sample	Glass, Teflon-lined cap	VOA, Metals, Semivolatiles, Pesticides/Herbicides - 14 days until extraction. Follow analytical protocol for aqueous holding time or holding time from leachate preparation.	Not specified	According to requested analysis
Water Reactivity	Approximately 100 ml for liquid sample Approximately 50 grams for solid sample	-----	None	14 day	-----

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	14 days	Method 1010 Pensky-Martens Closed-Cup Method Method 1020 Setaflash Closed Cup Method
Corrosivity	100 - 500 ml	Plastic	None	None	Method 1110 Corrosivity Toward Steel
California Assessment Manual CAM/STLC	Approximately 200 ml for liquid sample 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	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	100-200 ml for liquid sample	Plastic or Glass	HNO_3 to pH <2 for total	28 days	EPA 245.1 for water or EPA 7470A
Mercury, Total	Approximately 5 grams for solid sample		Filter on site, HNO_3 to pH <2 for dissolved		EPA 7471A for sediment Cold Vapor Method
Metals, Total Metals, Dissolved	300 ml for liquid samples Approximately 10 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 for total Filter on site, HNO_3 to pH <2 for dissolved	6 months	Flame AA-- See Individual Metal Methods Emission AA-- See Individual Metal Methods Graphite Furnace AA- See individual Metal Methods or ICP-- 200.7 or 6010

**** For individual metals the aggregate minimum volume is determined by the number of discrete analytical methods not the sum of all the individual analytes.**

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 Approximately 50 grams for solid samples	Plastic or Glass	Cool, 4 C	24 hours Extracted within 7 days, analyzed within 24 hrs. of extraction.	EPA 218.4 or EPA 7196A EPA 218.5 or EPA 7197 Extraction/AA Method EPA 7196 Colorimetric Method
Aluminum (Al)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 202.1, Flame EPA 202.2 or EPA 7020, Furnace
Antimony (Sb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 204.1 or EPA 7040, Flame EPA 204.2 or EPA 7041, Furnace
Arsenic (As)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 206.3 or EPA 7061, Hydride AA EPA 206.2 or EPA 7060, Furnace AA
Barium (Ba)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 208.1 or EPA 7090, Flame AA EPA 208.2, Furnace AA
Beryllium (Be)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 210.1 or EPA 7090, Flame AA EPA 210.2A or EPA 7091, Furnace AA
Boron (B)	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic only	Cool, 4° C	6 months	Curcumin Colorimetric EPA 212.3

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

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
Cadmium (Cd)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 213.1 or EPA 7130, Flame AA EPA 213.2 for EPA 7131, Furnace AA
Calcium (Ca)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 215.1 or EPA 7140, Flame AA
Chromium (Cr)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 218.1 or EPA 7190, Flame AA EPA 218.2 or EPA 7191, Furnace AA EPA 218.3 or EPA 7198, Chelation Extraction
Cobalt (Co)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 219.1 or EPA 7200, Flame AA EPA 219.2 or EPA 7201, Furnace AA
Copper (Cu)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 220.1 of EPA 7210, Flame AA EPA 220.2, Furnace AA
Gold (Au)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 231.1, Flame AA EPA 231.2, Furnace AA
Iron (Fe)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 236.1 or EPA 7380, Flame AA EPA 236.2, Furnace AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

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
Lead (Pb)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 239.1 or EPA 7420, Flame AA EPA 239.2 or EPA 7421, Furnace AA
Magnesium (Mg)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 242.1 or EPA 7460, Flame AA
Manganese (Mn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 243.1 or EPA 7480, Flame AA EPA 243.2 or EPA 7481, Furnace AA
Molybdenum (Mo)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 246.1 or EPA 7520, Flame AA EPA 246.2, Furnace AA
Nickel (Ni)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 249.1 or EPA 7610, Flame AA EPA 249.2, Furnace AA
Potassium (K)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 258.1, Flame AA
Selenium (Se)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 270.2 or EPA 7740, Furnace AA EPA 270.3 or EPA 7741, Hydride AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

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
Silver (Ag)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 272.1 or EPA 7760, Flame AA EPA 272.2, Furnace AA
Sodium (Na)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 273.1 or EPA 7770, Flame AA
Thallium (Tl)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 279.1 or EPA 7840, Flame AA EPA 279.2 of EPA 7841, Furnace AA
Tin (Sn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 282.1 or EPA 7870, Flame AA EPA 282.2, Furnace AA
Titanium (Ti)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 283.1, Flame AA EPA 283.2, Furnace AA
Vanadium (V)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 286.1 or EPA 7910, Flame AA EPA 286.2 or EPA 7911, Furnace AA
Zinc (Zn)**	100 ml for liquid sample Approximately 5 grams for solid samples	Plastic or Glass	HNO_3 to pH <2 Cool, 4° C	6 months	EPA 289.1 or EPA 7950, Flame AA EPA 289.2, Furnace AA

**** Each metal can also be analyzed by EPA 200.7, EPA 6010A, or EPA 6020.**

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	Flame AA EPA 215.1
Chloride	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA Method 325.3 Titrimetric Method
Copper**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 220.1
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	Flame AA EPA 236.1
Magnesium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 242.1
Manganese**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 243.1
Sodium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	Flame AA EPA 273.1
Sulfate	50 - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4 Turbidimetric
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	Flame AA EPA 289.1
Volatile Organics (VOAs)	2 - 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. INORGRANIC 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	EPA 206.3, Furnace AA
Barium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 208.1, Flame AA
Cadmium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 213.1 , Flame AA or EPA 218.2, Furnace AA
Chromium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 218.1, Flame AA EPA 218.2, Furnace AA
Lead**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 239.2, Furnace AA
Mercury	100 ml	Plastic or Glass	HNO_3 to pH <2	28 days	EPA 245.1 ,Cold Vapor
Selenium**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 270.2 ,Furnace AA
Silver**	100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 272.1 ,Flame AA
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 340.2, Ion Selective Electrode

**** 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, 4° C	14 days	EPA 305.1, Titrimetric
Alkalinity	50-100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 310.1 ,Titrimetric
Ammonia Nitrogen	100 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 350.1, Colorimetric
BOD	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 405.1
Boron	100 ml	Plastic	None	28 days	EPA 212.3 ,Curcumin
Chloride	100 ml	Plastic or Glass	None	28 days	EPA Method 325.3, Mercuric Nitrate or EPA 300.0, Ion Chromatography
COD	20 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA Methods 410.4, Colorimetric
Coliform Fecal Coliform	100 ml	Sterilized Plastic Bottles	Cool, 4° C $Na_2S_2O_3$ 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, 4° C	48 hours	EPA 110.2 or 110.3
Cyanide	500 ml	Plastic or Glass	Cool, 4° C NaOH to pH >12	14 days	EPA 335.3, Colorimetric
Electrical Conductivity	50 - 100 ml	Plastic or Glass	Cool, 4° 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, 4° C	48 hours	EPA 425.1, Colorimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Nitrate Nitrogen	100 ml	Plastic or Glass	Cool, 4° C	14 days	EPA 300.0, Ion Chromatography
Nitrate Nitrite Nitrogen	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	14 days	EPA 353.3, Cadmium Reduction
Nitrite Nitrogen	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 354.1, SPectrophotometric
Odor	200 ml	Plastic or Glass	Cool, 4° C	24 hours	EPA 140.1
Oil and Grease	1 liter	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 413.1, Gravimetric
Orthophosphate	50 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 365.2, Ascorbic Acid
pH	50 ml	Plastic or Glass	None	Immediate	EPA Method 150.1, pH Meter
Phenolics	500 ml	Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 420.1, 4-AAP
Radioactivity	1-18 liters	Plastic or Glass Glass only for Tritium	HCl or HNO_3 * to pH <2 *Some exceptions	-----	Standard Method 701
Silica	50 ml	Plastic	Cool, 4° C	28 days	EPA 370.1, Colorimetric Flame AA Method
Sulfates	50 ml - 100 ml	Plastic or Glass	Cool, 4° C	28 days	EPA 375.4, Turbidimetric or EPA 300.0, Ion Chromatography
Sulfides	500 ml	Plastic or Glass	NaOH to pH >9 2 ml Zinc Acetate Cool, 4° C	7 days	EPA 376.1 ,Titrimetric
Sulfites	50 ml	Plastic or Glass	None	Immediate	EPA 377.1 ,Titrimetric
TOC	25 ml	Plastic or Glass	Cool, 4° C H_2SO_4 to pH <2	28 days	EPA 415.2, TOC Analyzer
Total Dissolved Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.1, Gravimetric

H. GENERAL WET CHEMISTRY AND MISCELLANEOUS ANALYSIS (continued)

PARAMETER	MINIMUM VOLUME REQUIRED	CONTAINER	PRESERVATION	MAXIMUM HOLDING TIME	METHOD FOR ANALYSIS
Total Hardness	50-100 ml	Plastic or Glass	HNO_3 to pH <2	6 months	EPA 130.2, Titrimetric Standard Method 314-A Calculation
Total Kjeldahl Nitrogen	1 liter	Plastic or Glass	Cool, 4° C 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, 4° C 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 Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days, but this may vary between states so regulations must be consulted	WTPH-HCID by GC/FID or Modified Method 8015, GC
Total Petroleum Hydrocarbons as Gas, by GC	40 ml Approximately 20 grams for a solid sample	Glass	Cool, 4^0 C, Methanol preservation may be a requirement of some states so state regulations must be consulted Cool, 4^0 C	14 days	WTPH-G by purge and trap GC/FID or Modified Method 8015, GC/FID
Total Petroleum Hydrocarbons as Diesel, by GC	1 liter Approximately 50 grams for a solid sample	Glass	Cool, 4^0 C	14 days	WTPH-D by GC/FID or Modified Method 8015, GC/FID
Total Phosphate	50 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	28 days	EPA 365.4, Colorimetric
Total Residue	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.3 , Gravimetric
Total Settleable Solids	1 liter	Plastic or Glass	Cool, 4° C	48 hours	EPA 160.5, Imhoff Cone
Total Suspended Solids	100 ml	Plastic or Glass	Cool, 4° C	7 days	EPA 160.2, Gravimetric
Total Volatile Solids	100 ml	Plastic or Glass	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 160.4, Gravimetric
TOX	500 ml	Amber Glass, Teflon Septum	Cool, 4° C Add H_2SO_4 to pH <2	7 days	EPA 450.1 or EPA 9020, TOX Analyzer
Turbidity	100 ml	Plastic or Glass	Cool, 4° C	48 hours	EPA 180.1

APPENDIX 9

Checklist For Analyses With Short Holding Times

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)

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-4103

Title: Reviewing a Sample Delivery Group (SDG) and Updating Projqc in the LIMS

Revision History:

Number	Date
0	4/09/96

Written by: Diana Spence Date: 4/9/96
Diana Spence

Reviewed by: Harry Romberg Date: 4-9-96
Harry Romberg, QC Officer

Approved by: Karen J. Kotz Date: 4/9/96
Karen Kotz, Laboratory Director

UNCONTROLLED

Table of Contents

1. INTRODUCTION AND SCOPE 3

1.1. METHOD DESCRIPTION 3
1.2. DEFINITION OF TERMS 3

2. EQUIPMENT LIST 3

2.1. EQUIPMENT 3

3. OPERATION PROCEDURES 3

3.1. REVIEWING THE SDG ENTRY IN LIMS 3
3.2 ENTERING A WORK ORDER INTO 'PROJQC' OF LIMS 5
3.3 CREATING A NEW PROJQC 6

APPENDIX I 9

EXAMPLE OF SDG ENTRY IN LIMS 9

APPENDIX II 10

EXAMPLE OF PROJQC IN LIMS 10

APPENDIX III 11

EXAMPLE OF SDG PAPERWORK 11

1. Introduction and Scope

1.1. Method Description

1.1.1 The project manager (PM) or their designee reviews the LIMS computer entry versus the chain-of-custody (COC) for accuracy. This should be done as soon as practical and within twenty-four hours (24) of sample receipt.

1.1.2 This method is restricted to use by the person (usually a project manager) who performed the work order transmittal.

1.2. Definition of Terms

1.2.1. SDG - Sample Delivery Group

Projqc - Project QC section of the LIMS. This is where all of the work orders for a specific project are listed with additional information about the QC requirements, TATs and deliverables.

2. Equipment List

2.1. Equipment

2.1.1. The person performing this procedure must have access to a computer which is connected to the LIMS.

3. Operation procedures

3.1. Reviewing the SDG Entry in LIMS

3.1.1 Once a work order has been transmitted, go to the SDG section of the LIMS. This is done by entering [SDG, space, SDG name] at the J(1): prompt. The SDG name is found in the fractions sections of the work order in the SDG field labeled 'SDG#'. **Example** - J(1): SDG HCST4. See Appendix I for an example.

3.1.2 In the SDG verify that all the samples have been entered and that the correct 'fraction' of the SDG (I,G, V, P, etc.). The letters refer to the type of analysis, i.e., Inorganics, GC, Volatiles, and PAHs. These are a few of the types of

fractions which might be contained in a SDG. It is not important to know the abbreviations. However, it should be verified that all analyses for each sample can be found in the SDG by looking at the various fractions for that SDG. A fraction may contain more than one analysis. For instance an 'I' fraction (inorganics) might have TOC, Metals and TSS.

3.1.3 When the SDG name is entered at the J(1): prompt, the 'first' page of the first fraction of a SDG will be displayed. The first fraction is determined alphabetically based upon the letter associated with an analysis. For instance, if a work order has analyses for GC analysis (G), metals (I) and PAHs (P), the fractions of a SDG would be listed in LIMS with GC analyses first, metals second, and PAHs last. The information contained on the first page is:

- Laucks sample ID
- QC Designations
- Client ID
- Matrix

3.1.4. Verify that all the samples for that fraction (analysis) have been entered. this is facilitated by entering [F6] which displays the second , third and fourth pages of a fraction. [F6] is a toggle key which pages through the four screens of each SDG entry. The far left hand column of the computer screen will always display the lists of the SDG names with the fractions appendix letter. **Example** - GSI01G (GC fraction), GSI01I (inorganics fractions). See pages 1,2 and 3 of Appendix 1 for examples of each page of a SDG.

3.1.5 The second page of a SDG contains the Laucks samples ID, the VTSR (Verified Time of Sample Receipt) and date collected information.

3.1.6 The third page of a SDG contains the Laucks sample ID and the specific analysis requested. It is important to review this page carefully. If there are many samples in that fraction, they may not all be visible at once. Move down the screen to view all samples by repeatedly pushing the down arrow key, [↓].

3.1.7 Once this fraction has been checked, the other fractions can be checked by using the [F3] (moves the cursor up) and [F4] (moves the cursor down) function keys to place the cursor on the next fraction of the SDG. It is displayed in the column on the left of the screen.. It is possible to view the same page of different fractions by just moving the cursor to the next fraction. For example, if you are in page three of a fraction, when you move to the next fraction, page three of this fraction will

be displayed. **[F6] controls the page selection of the SDG, [F3] and [F4] respectively in the far left column.**

3.1.8 If corrections are necessary, return the COC and log-in documents to the Sample Control department. If the person performing the review makes the corrections, the changes must be saved by entering [F8].

3.1.9 To exit the SDG, enter [F1] until the J(1): prompt is displayed.

3.2 Entering a Work Order into 'Projqc' of LIMS

3.2.1 At the J(1): prompt, enter [projqc, space, projqc name]. Example - J(1): projqc OHM_Hawaii. If you are not sure of the projqc name, a name may be entered which is similar. This will bring up the projqc in the vicinity of the name which is desired. The cursor is moved up, [F3], or down, [F4}, to the desired name. The projqc names will be displayed in the far left column of the screen. See page 1 of Appendix II.

3.2.2 Once the correct project name is displayed, move the cursor down to the next empty line in the work order column. enter the work order number. The SDG will appear in the SDG list column. Move the cursor to the matrix columns (W = Water, S = Soil, O = Oil). Under the appropriate column, enter the number of samples for each matrix. If the SDG is to remain open, no other information should be added. Enter [F8] to save the updated SDG.

3.2.3 It the SDG is to be closed, the due date for the data to be submitted to the reporting department and the due date for the hardcopy report to the client must be entered into the appropriate columns. These dates are displayed in Projqc as 'Office' and 'Client' respectively. See page 1 of Appendix II. Enter [F8] to save these changes.

3.2.4 SDG closure is determined by several factors:

- The number of samples in the SDG, per the EPA definition, should not contain more than twenty samples of the same matrix received over a period of not more than 14 calendar days.

- If it is known that more samples will be arriving for the same project in less than fourteen days, and the SDG is not full, it may be desirable to maintain the SDG in 'open' status until arrival of the next samples.

- If the client desires a fast TAT for the hardcopy package, the SDG should be closed regardless of the number of samples in it.

These are judgment decisions with the exception of the EPA definition of an SDG), which must be made by the project manager.

3.2.5 Once the SDG has been closed, send e-mail notification of the closure to all departments affected. This would be any department involved with the analyses for the work order, Sample Control and the reporting department.

3.2.6 Finally, the paperwork is submitted to the reporting department detailing the nature and status (open or closed) of an SDG. The forms submitted would be the following, arranged in the order listed below:

- Pre-package checklist
- Chain-of-Custody forms (original or top, white copy)
- * Sample Receipt Log (1) CLP (original)
- * Supplemental Sample Receipt Log (original)

* A copy of each of these forms must be made and attached to the copy of the COC for the transmittal.

See Appendix III for examples of these forms.

3.3 Creating a New Projqc

3.3.1 When a new project commences, it may be necessary to create a new entry in Projqc. The factors which would determine if this is necessary may include the following:

- The complexity of the project
- The duration of the project
- How many laboratory departments are affected by the project

Projqc entries are applicable to both CLP and non-CLP project. The information contained in this section of the LIMS is accessible by all laboratory staff who have rights to the LIMS and is a valuable form of communication for project requirements. However, it does not take the place of kick-off meetings for projects or e-mail regarding specific samples problems.

3.3.2 To create a new projqc entry, enter [projqc] at the J(1): prompt. This will display the first projqc entry in the LIMS. The cursor will be in the field called 'Proj Name:'. Enter the name of the new projqc entry. this name may consist of up to ten characters, alpha and numeric, and one underscore in the place of a hyphen, if desired. Once entered, push [Enter]. The cursor will move to the next field, 'Client Code'. See page 1 of Appendix II.

3.3.3 In the 'Client Code' field, enter the correct client code for this work order. This can be found in the work order. Push [Enter]. The cursor will next appear in the 'Desc:' field.

3.3.4 In this field enter a brief description (one cryptic sentence) of the project. Push [Enter]. the cursor will next appear in the bottom line of the projqc header. Enter the correct information for these questions (QAPP?, pH/Temp?). This would usually be 'yes' if the project is CLP or USACE (US Army Corps of Engineers).

3.3.5 Due to software complications, in the field 'Sort by (W/S):' it is best to enter [N].

3.3.6 Enter [F8] to save this new projqc. Item 3.3.1 through 3.3.5 would be considered the header information for a projqc. This is page 1 of the projqc. It is here that the work orders for that project will be entered when the samples arrive at the laboratory.

3.3.7 Next go to page 2 of the projqc. This is accomplished by pushing [F6]. The header information will already be there. The information which must be entered here is:

- Results TAT (Fax)
- Report TAT (hardcopy)
- Deliverables - CLP, LTL (Laucks Testing Laboratory or non-CLP)
- MPR - Monthly Progress Report (required for HAZWRAP projects)
- EDD deliverables - CLP or LTL (any non-CLP, custom EDD)
- Deliverables Comments - Any special project comments
- Penalties - enter any applicable penalties here

Enter [F8] to save this information.

3.3.8 Next go to page three of the projqc. This is accomplished by entering [Ctrl,F6]. of a project can be written as an overview. See page three of Appendix II. The information contained here is:

- Client name
- Project Name
- Overview
- Schedule
- Analytes
- Protocol
- QC
- Turnaround
- Penalties
- CRDLs
- Holding Times
- Deliverables
- Additional Comments

After entering this information, enter [F8] to save it. It may be advantageous to enter [F8] periodically as the information is entered to prevent loss in the event of a power surge.

Appendix I

Example of SDG Entry in LIMS

GSI01
KEYS
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P
GSI02V
GSI03G

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/95
Fraction : G SPVMITO Updated: 10/23/95
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
PROJQC ID : GS_CHANUTE Max. Samps: 21

Work Ord	Samp Num	QC	Client ID	Mat	TS	pH	Associated Blank
9510423	01		B952/SB-952-1/SS4,6-8/LK	S	88		B1018GSVSLW
9510423	02		B952/SB-952-1/SS9,16-18/LK	S	89		B1018GSVSLW
9510423	03		B952/SB-952-2/SS4,6-8/LK	S	83		B1018GSVSLW
9510423	04		B952/SB-952-2/SS5,8-10/LK	S	84		B1018GSVSLW
9510434	01		B952/SB-952-10/SS8,14-16LK	S	84		B1018GSVSLW
9510434	02		B952/SB-952-3/SS3,4-6/LK	S	84		B1018GSVSLW
9510434	03	MS	B 52/SB-952-3/SS6,10-12/LK	S	89		B1018GSVSLW
9510434	05	BMS	CH-5092500-S-SPOON-ERB-1	W			B1017GSVWLQ2
9510434	06		CH-POT-FB-1	W			B1017GSVWLQ2
9510434	07		CH-ASTM-5092500-FB-1	W			B1017GSVWLQ2
9510459	01		B952/SB-952-4/SS4,6-8/LK	S	84		B1018GSVSLW
9510459	02		B952/SB-952-4/SS7,12-14/LK	S	89		B1018GSVSLW
9510459	03		B952/SB-952-5/SS4,6-8/LK	S	87		B1018GSVSLW
9510459	04		B952/SB-952-5/SS6,10-12/LK	S	87		B1018GSVSLW
9510459	08		B952/SB-952-6/SS4,6-8/LK	S	84		B1018GSVSLW
9510459	09		B952/SB-952-6/SS6,10-12/LK	S	90		B1018GSVSLW
9510497	01		B952/SB-952-7/SS3,4-6/LK	S	89		B1018GSVSLW
9510497	02		B952/SB-952-7/SS8/4-16/LK	S	91		B1018GSVSLW
9510540	01		B952/SB-952-8/SS3,4.0-6.0	S	90		B1018GSVSLW
9510540	02		B952/SB-952-8/SS6,10.0-12	S	90		B1018GSVSLW

SDG Database Report
Page 2

Laucks Testing Labs Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	VTSR	Date Collected
9510423-01		10/11/95	10/10/95
9510423-02		10/11/95	10/10/95
9510423-03		10/11/95	10/10/95
9510423-04		10/11/95	10/10/95
9510434-01		10/12/95	10/11/95
9510434-02		10/12/95	10/11/95
9510434-03		10/12/95	10/11/95
9510434-05		10/12/95	10/10/95
9510434-06		10/12/95	10/10/95
9510434-07		10/12/95	10/10/95
9510459-01		10/13/95	10/11/95
9510459-02		10/13/95	10/11/95
9510459-03		10/13/95	10/11/95
9510459-04		10/13/95	10/11/95
9510459-07		10/13/95	10/12/95
9510459-08		10/13/95	10/12/95
9510459-09		10/13/95	10/12/95
9510497-01		10/14/95	10/12/95
9510497-02		10/14/95	10/12/95
9510540-01		10/17/95	10/15/95
9510540-02		10/17/95	10/15/95

SDG Database Report
Page 3

Laucks Testing Labs

Report Date: 01/25/96

SDG Group : GSI01 Date Due: 11/10/95
Fraction : G
Project : AFCEE/Chanute 952 Client: GSI (TPH 8015) JP4
SAS Number: _____ Case Number: _____

Work Ord	Samp Num	Fractions JP4										
9510423-01		X										
9510423-02		X										
9510423-03		X										
9510423-04		X										
9510434-01		X										
9510434-02		X										
9510434-03		X										
9510434-05		X										
9510434-06		X										
9510434-07		X										
9510459-01		X										
9510459-02		X										
9510459-03		X										
9510459-04		X										
9510459-07		X										
9510459-08		X										
9510459-09		X										
9510497-01		X										
9510497-02		X										
9510540-01		X										
9510540-02		X										

Comments:

G
KEYS
GLS08O
GLS08P
GLS08S
GLS08T
GLS08V
GLS09E
GLS09I
GLS09O
GLS09P
GLS09S
GLS09T
GLS09V
GSI01G
GSI01I
GSI01P
GSI01V
GSI02G
GSI02I
GSI02P

SDG Database

SDG Group : GSI01 Date Due: 11/10/95 Created: 10/12/95
Fraction : I SPVMITO Updated: 10/17/95
Project : AFCEE/Chanute 952 Client: GSI (Inorganic)
PROJQC ID : GS_CHANUTE Max. Samps: 21

Comments:
Use Control F6 for more PROJQC details.

F7=Del,/n,Name 8=Write,/n,In_use 9=Print,Db_enter?,Keys? 10=More,Help,Ke

Appendix II

Example of Projqc in LIMS

OHM
KEYS
HC_MANANN
'C_MANANNS
.IC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/9
Desc: UST sites Updated: 01/22/9
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Ord#	Last Date	SDG#	Office	Client	Matrix W	Matrix S	Matrix O
9512516	12/19/95	HI033	01/31/96	02/02/96	2	3	
9601349	01/22/96	HI034	02/20/96	02/22/96	1	7	
9601351	01/10/96	HI035			3		
9601587	01/22/96	HI034	02/20/96	02/22/96	2	6	

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

OHM
KEYS
HC_MANANN
HC_MANANNS
HC_MANANNW
KIC_ARCO
LOW_AFB_IN
LOW_AFB_OR
MCCHORDAFB
MCCHORDLTM
METRO_TBT
OHM-HAWAII
OHM-SITEA
OHM-SITEF
PASCO_AIR
PASCO_P
PASCO_S
PASCO_W
Q_CITY_F
SKAGIT
SODASPRING

Project QC Requirements Database

Proj Name: OHM-HAWAII Client Code: OHM_HAWAII Created: 12/19/95
Desc: UST sites Updated: 01/22/96
QAPP? Y pH/Temp? Y Sort by (W/S): N By : DIANA

Results turnaround 7 WORK.DAYS
Report turnaround 30 CAL.DAYS

Deliverables	Penalties
LTL report	%
"CLP" report X	%
MPR required	%
CLP disk	%
LTL disk	%
Other	%
Other	%
	%

Deliverables comments

F7=Del,,Name 8=Write,,In_use 9=Print,Help menu,PrtSc 10=More,Help,Key

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Client : OHM Remediation Services, Inc.

Project Name: UST Sites in Hawaii

Overview : 90% project consists of Soil samples, 10% water/rinsate samples

Schedule : Delivery of samples over 6 months. Weekly schedule will be obtained one week in advance of sample receipts.

Analytes : Water
29- BTE samples via 8020
36- 8015 Mod (TPH gas or diesel or 418.1)
17- 6010 for Pb, Cd, Cr
30- 8310 Napnthalene, Acenapnthene, Fluoranthene, and low level Benzo(a)pyrene
7- 8080 Low level PCBs 2L to 1mL
7- 8010 tetrachloroethylene, and 111-trichloroethane
10- 1020 flammability
1- HPLC (method supplied by client 2/16) for PGDN and 2-nitrodiphenylamine, Samples to arrive after March
5- 6010 Pb only

Soil
260- BTE samples via 8020
327- 8015 Mod (TPH gas or diesel or 418.1)
152- 6010 for Pb, Cd, Cr
264- 8310 Naphthalene, Acenaphthene, Fluoranthene, and Benzo(a)pyrene
60- 8080 PCBs
7- 8010 tetrachloroethylene, tand 111-trichloroethane
10- 1020 flammability
3- HPLC (method supplied by client 2/16) for PGDN and 2-nitrodiphenylamine, Samples to arrive after March
40- 6010 Pb only

Protocol : SW-846 with NEESA Level C data package. See me if you need a copy of the NEESA Requirements. I have the June 88 version. Site specific QC required. Batch in SDGs.

QC : Yes! See handout submitted to;
JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,SO

Turnaround : FAX 7 working days from sample receipt Hard Copy 30 calendar days from SDG closure EDD Not applicable

File: Y:OHM-HAWAII.PCC
Date: 01/24/96

Penalties : See PROJQC, page 2

CRDLs : Yes! See minimum detection limits in handout submitted to;
JMB,MN,Bill,BarbM,MC,CD,MS,MIK,PJ,TM,SO

Holding Time: Routine SW-846 from collection.

Deliverables: NEESA Level C

Comments :

All stock pile samples must be thoroughly homogenized prior to analysis.

UPDATES will be added (with the date) to this file.

Pam, see MarkS regarding special test codes for low level metals prep! Something like, LXWM1 for the digestion. Also, these samples will be batched in SDGs. See MarkS.

First tentative schedule starting 3/2 or 3/3:

90 soils - BTE (8020) + X
40 soils - TPH Gas
50 soils - TPH Diesel OR 418.1
50 soils - 8310 PAHs
40 soils - Pb only (6010)

3/15/95 sh;

Art Taddeo requested pricing for TCLP Pb, Cd, and Cr (to comply with TCLP regulations). I provided pricing for soils at $130.00 ea.

10/26/95 ds

Data packs are to be sent to:

Kim Osgood
OHM
20015 72nd Ave.S
Kent, WA 98032

Reports are to be faxed to Bob Rooks at 808-682-1880 in HI.

Appendix III

Example of SDG Paperwork

PRE-PACKAGE CHECKLIST

SDG# GTL27 open ☐ closed ☑

Work Order #(s)	Fax Due Date(s)
960512	1/16/96

Due to Office: 2/7/96

Due to client: 2/9/96

E.D.D.: yes ☐ Custom or CLP *(circle one)* no ☑

If yes, can the package ship without the disk? _____

Full Deliverables? yes ☑ no ☐

If no, what forms/raw data are required? _____

No. of Copies: 1

Special package requests:

Assigned Document controller: _____

THIS INFORMATION WILL BE USED FOR REPORTING/BILLING* (SEE BELOW)

NAME: Groundwater Technology GSI

ADDRESS: 19053 W Valley Hwy D-104
Kent WA 98302

ATTENTION: Diana Spence

PROJECT NAME: Bangor Site A

PROJECT CONTACT: Stan Thompson

TELEPHONE/FAX: 360 779 9564/ 360-779-1676

JOB/P.O. NO.: 83001-2153 110

SAMPLER (SIGNATURE): (PRINTED NAME) Daryl Brown

CHAIN OF CUSTODY RECORD: 1515

WORK ORDER ID: 83-01-2153 PAGE 1 OF 1

DATE 1-8-96

SUBMITTED AT:

Laucks Testing Laboratories, Inc.

TESTS TO PERFORM: M8330

LAB SAMPLE	SAMPLE ID / LOCATION	DATE	TIME	NO. OF CONTAINERS	OBSERVATIONS, COMMENTS, SPECIAL INSTRUCTIONS
1)	INF-CL-209-010896	1-8-96	0900	2L	Fax Results to 360-779-1676
				2L	

INSTRUCTIONS
1. USE ONE LINE PER SAMPLE.
2. BE SPECIFIC IN TEST REQUESTS.
3. CHECK OFF TESTS TO BE PERFORMED FOR EACH SAMPLE.

*BILLING INFORMATION, IF DIFFERENT THAN ABOVE
NAME
ADDRESS
ATTN:
CITY, STATE, ZIP

TURNAROUND REQUEST
- [ ] 24-48 HRS (100% SUR)
- [x] 5 DAYS (50% SUR)
- [ ] STD. 10-11 DAYS
- [ ] OTHER

TOTAL NO. OF CONTAINERS 2L

CHAIN OF CUSTODY SEALS? [ ] YES [ ] NO [ ] NA

SHIPPED VIA [ ] UPS [ ] FED EX [ ] BUS [ ] HAND [ ] _____

TEMPERATURE _____ [ ] AMBIENT [ ] REFRIGERATED

RELINQUISHED BY (SIGN AND PRINT)	DATE TIME	RECEIVED BY (SIGN AND PRINT)	DATE TIME
Daryl Brown	1-8-96 1600	Pony Express	
		PAM R. JOHNSON Laucks Testing Labs	1/9/96 0930

Laucks Testing Laboratories, Inc.
SAMPLE RECEIPT LOG (1) CLP

?:PM\Holly\coc\taxmemo.doc

Initial once samples are checked in _____

DATE RECEIVED: 1/9/96
TIME RECEIVED: 0726
CLIENT NAME: GSI
SDG #: GTE27
COC #: N/A

SAMPLE LOG-IN DATE: 1/9/96
WORKORDER #: 9601313
CLIENT PROJECT: Bangor Site A
AIRBILL ATTACHED?:(#) 2157 6393
RECEIVED BY: B

Non-Conformance: (Check applicable item(s)) Client IDs affected:

- ☐ (1) Not enough sample sent for proper analysis. #s affected: _____
- ☐ (2) Sample Bottle received broken and/or cap not intact _____
- ☑ (3) Custody seal: Absent ____ Present/Intact ✓ Present/Broken _____
- ☐ (4) Any temperature out of compliance: _____
- ☐ (5) Sample received outside of holding time. _____
- ☐ (6) Sample not properly preserved. pH = ___. Wrong preservative used. _____
- ☐ (7) Illegible sample numbers or label missing from bottles. _____
- ☐ (8) Identification on bottle same as identification on paperwork: yes: _____ no: _____
- ☐ (9) Incomplete instructions received with sample(s), i.e., no Request for Analysis, no Chain-of-Custody. _____
- ☐ (10) Samples received in improper container. _____
- ☐ (11) Samples held in field before receipt by Lab. Days (specify) _____
- ☐ (12) Air Bubble(s) in ___ of ____ samples for volatiles analysis. _____
- ☐ (13) Other _____

CORRECTIVE ACTION: (Check applicable item(s))

	Correction action taken by: Initials	Date
☐ (1) Client informed verbally (Client Services).	_____	
☐ (2) Client informed by memo/letter/fax (Client Services).	_____	_____
☐ (3) Sample processed "as received" (Sample Entry).	_____	_____
☐ (4) Re-sampling requested of client (Client Services).	_____	_____
☐ (5) Samples placed "on hold" until further notice (Sample Entry/Client Services).	_____	_____
☐ (6) NOTE IN NARRATIVE. See temperature/pH login sheet. (Sample Entry).	_____	_____
☐ (7) Other (Specify) _____	_____	_____

* When complete (within 24 hours of nonconformance) forward to QA. Original to be forwarded to initiator to be included in transmittal file.

Comments:

Laucks Testing Laboratories, Inc., Supplemental Sample Receipt Log

Work Order Number: 9601313
Assigned SDG Number: [illegible]

Temperature	pH or Bottle Types								
	8330								
1 2 0 1 2 3	2) 7								

Allowable temperature and pH ranges (neutral pH defined as a value between 5 and 9)

emperature	Allowable temperature range is 4± 2 degrees Celsius
cid Preserved pH	pH must be less than 2
ase Preserved pH	pH must be greater than 12

TEMPLOG.DOT : [illegible]

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: _____ Date: 1-31-96
Tom Marino

Reviewed by: _____ Date: 1-31-96
Charlene Nix

Reviewed by: _____ Date: 1-31-96
Harry Romberg, QC Officer

Approved by: _____ Date: 1/31/96
Karen Kotz, Laboratory Director

UNCON[illegible]D

Table of Contents

1. Introduction and Scope 3
    1.2 Complete CLP-type data package (level IV): 3
    1.3 Forms only data package (level III): 3
    1.4 Database Report (level II): 3
    1.5 Paper Job Report (level I): 3
2. Description of Contents of Level III and IV packages 3
    2.1 Cover Page 4
    2.2 Narrative 4
    2.3 Chain-of-Custody Copies: 5
    2.4 Index: 5
    2.5 Organic fractions 5
    2.6 Inorganics Fractions: 8
Appendix I 10

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.

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:

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.

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:

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

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

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.

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.

Laucks Testing Laboratories
Metals
Levels of Reporting

	Level I	Level II	Level III	Level IV = CLP
Narrative			Y	Y
Chain-of-Custodv	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.

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

Y indicates that this element is present in the hardcopy delivered to the client.

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

Controlled Document

No. 20 Assigned to: TETRA

Table of Contents

1. Introduction and Scope 3
   1.1 Method Description 3
   1.2 Definitions 3
2. Equipment List 3
   2.1 Standard Laboratory Glassware 3
3. Safety precautions and Waste Disposal 3
   3.1 Safety Precautions 3
4. Operation procedures 4
   4.2 ALKALINITY 4
   4.3 AUTO ANALYZER (refer to the applicable analytical SOP) 4
   4.4 BOD 4
   4.5 CYANIDE 4
   4.6 HEXAVALENT CHROMIUM 4
   4.7 FORMALDEHYDES 5
   4.8 HARDNESS 5
   4.9 KJELDAHL NITROGEN-LOW LEVELS (TKN AND AMMONIA) 5
   4.10 OIL AND GREASE – Method 1664 5
   4.11 METALS (INCLUDING MERCURY) 5
   4.12 NITROCELLULOSE 6
   4.13 PHENOL 6
   4.14 PHOSPHATE 6
   4.15 TOC (SOILS) 6
   4.16 TOX 6
   4.17 TOTAL SOLIDS 6
5. References 7

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.

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.

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.

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

5. References

5.1.1 See the applicable analytical or preparation SOP for specific cleaning issues and references.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

Method #: LTL-7015

Title: **Acid Digestion of Sediments, Sludge, and Soils by SW846 Method 3050B**

Revision history:

Number	Date
3	06/20/05
2	10/23/02
1	3/20/00
0	1/18/99

Revised by: Bill Ambacher Date: 6/21/05
Bill Ambacher, Inorganic Supervisor

Approved by: Harry Romberg Date: 6/21/05
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 6/22/05
Kathy Kreps, Laboratory Director

Controlled Document
No. 20 Assigned to: TETRA

Table of Contents

1. INTRODUCTION AND SCOPE 3
2. DEFINITION OF TERMS 3
3. SUMMARY OF METHOD 4
4. SAFETY PRECAUTIONS AND WASTE DISPOSAL 4
5. EQUIPMENT 5
6. REAGENTS 6
7. ACID DIGESTION PROCEDURE 6
8. QUALITY CONTROL 7
9. REFERENCES 8
APPENDIX A 9
DIGESTION BENCHSHEET AND LOG 9
APPENDIX B 11
CHECKLIST FOR 3050B 11
APPENDIX C 13
FLOWCHART 13
APPENDIX D 15
QUALITY CONTROL SOLUTIONS 15

Controlled Document
No. _____ Assigned to: _____

1. Introduction and Scope

1.1. This method is an acid digestion procedure used to prepare sediments, sludges, and soil samples for inductively coupled argon plasma spectroscopy (ICP) or inductively coupled argon plasma spectroscopy - mass spectrometry (ICP-MS). Samples prepared by this method may be analyzed by ICP and ICP-MS for all the listed metals: Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and Zn.

1.2. Although Arsenic, Selenium and Tin are not listed in the original SW846 document as analytes that may be analyzed from samples prepared by Method 3050B, Laucks has demonstrated adequate recovery for these analytes from this digestate.

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.

1.4. This method requires the addition of hydrogen peroxide, which contains a tin stabilizer. Because of this, the reporting limit for tin should be no lower than 10 mg/Kg. Any tin found in the preparation blank should be less than ½ of this reporting limit for samples to be reported.

1.5. Sample Collection, Sample Storage, Holding Times

1.5.1. There are no storage conditions for metals in soil, but typically samples should be collected in plastic or glass bottles. Samples are cooled to 4 ± 2° C at time of collection. Samples are kept at 4 ± 2° C until digestion. Holding time is 6 months based on time of collection.

2. Definition of Terms

2.1. 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.

2.1.1. **Blank Spike** – A background free matrix (DIW for water, clean sand for soils/sediments) to which known target are added each time samples are prepared. Blank Spikes are required on all AIMTech (formerly HAZWRAP) and NFESC work.

2.1.2. **Batch Identifier** - A number preceded by an "A" that indicates the date of analysis or preparation, the instrument used to acquire the data, the analyte determined, and the run number (i.e. A111694ICP01 indicates the first run using the ICP on November 16, 1994).

2.1.3. **CLP** – Contract Laboratory Program – The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

2.1.4. **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

2.1.5. **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.

2.1.6. **QC period** - Quality control period - An analysis sequence initiated by the analysis of one or more.

3. Summary of Method

3.1. A representative 1.0 to 1.5g sample (larger quantities may be needed to meet specific client requirements) is digested in nitric acid and hydrogen peroxide. The digestate is then either reduced to a low volume or refluxed with hydrochloric acid. Dilute hydrochloric acid is used as the final reflux acid for the analysis of Ag, Al, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Tl, V, and Zn. The diluted samples have an approximate acid concentration of 5% (v/v).

4. Safety Precautions and Waste Disposal

4.1. Safety Precautions

4.1.1. These procedures involve the use of strong and/or hot acid solutions. The analyst must wear eye protection, a lab coat, and gloves to protect against burns.

4.1.2. These procedures involve hot plates which may present the danger of burns from heated surfaces and electrical hazards. The analyst must take appropriate caution to avoid injury from these sources.

4.1.3. Samples and spiking solutions may contain high levels of toxic metals and other unknown constituents. The analyst must take every precaution to avoid contact with these potentially hazardous materials and should wash hands thoroughly before taking any breaks, eating, or going home for the day.

4.1.4. Fertilizer samples are chemically complex matrices and should be handled with extreme caution. Many components found in fertilizer are highly reactive to acid and all acid additions should be made with special care, in a hood, standing back from the sample. Also, these samples have been known to "pop" or explode during heating, so care should be taken not to stand close to or have any body part over the samples while they are being heated.

4.2. Waste Disposal

4.2.1. Digestates are neutralized and then disposed down the sink. A record of the amount of neutralized waste is kept by the disposal sink.

4.2.2. Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal.

5. Equipment

- 250-mL Erlenmeyer flasks or equivalent, acid-washed
- Analytical balance capable of accurately weighing to the nearest 0.01 g
- Reflux caps fitted for Erlenmeyer flasks, acid-washed
- Eppendorf or other micropipets-refer to SOP LTL-9003 for calibration procedures.
- Hot plate or MOD Block, adjustable and capable of maintaining a temperature of 90-95° C
- 100-mL graduated cylinder, acid-washed
- Filter paper, Whatman No. 41 or equivalent
- Glass funnels, acid-washed
- 100-mL volumetric flasks, acid-washed
- 100 mL sample digestate bottles, acid-washed
- Thermometer

Note: All glassware used for this digestion must be prepared by the method stated in Inorganics Glass Cleaning Procedures, SOP #: LTL-7003.

6. Reagents

6.1. Reagent Water. Reagent water will be interference-free deionized water. All references to water in the method refers to reagent water unless otherwise specified.

6.2. Nitric acid (concentrated), HNO_3, ACS Reagent grade or better

6.3. Hydrochloric acid (concentrated), HCl, ACS Reagent grade or better

6.4. Hydrogen peroxide (30%), H_2O_2, ACS Reagent grade or better.

7. Acid Digestion Procedure

- See Appendix A for digestion logs.
- See Appendix B for flowchart.
- See Appendix C for Quality Control solutions
- See Appendix D for Procedure Checklist

7.1. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh 1.0-1.5 g of sample to the nearest 0.01 g directly into an Erlenmeyer flask or digestion vessel. For samples with low percent solids or if dry-weight equivalent is required, a larger sample size may be used to attain the equivalent of 1.00-1.50 g dry basis as long as digestion is completed. A larger sample size may also be needed for specific client requests. Prior to the addition of reagents, appropriate spiking solutions should be added to the blank spike and matrix spike(s). If an analyst has less than 6 months experience, a spiking witness must be present. Record weight, sample description, and bottle number on the digestion log (Appendix I).

7.2. Place the reflux caps on the flasks, add 5 mL of concentrated HNO_3, 5 mL H_2O and mix the slurry. Heat the samples to 95° C and reflux without boiling for 15 minutes. Allow the samples to cool. Record the temperature in the digest log. Add 5 mL of concentrated HNO_3, heat 30 min and allow the samples to cool. If brown fumes are generated, continue to add acid, heat and cool until fumes are no longer emitted. Reflux the samples for two hours or allow them to evaporate to 5 mL with reflux caps. Allow the samples to cool.

7.3. Add 2 mL water, 3 mL 30% H_2O_2, heat for 10 minutes and cool the samples. Add 1 mL 30% H_2O_2, reflux for 10 minutes and allow the samples to cool. Repeat the addition of 1 mL H_2O_2 and heat until effervescence is minimal or a maximum of 10 mL H_2O_2 is added. Make sure that sample losses do not occur due to excessive effervescence. Reduce the sample to 5 mL.

7.4. For ICP analysis only-Add 10 mL of concentrated HCl. Heat the sample for 15 minutes and allow it to cool.

7.5. Dilute the sample to 100 mL with water in a graduated cylinder and place in an acid-washed digestate bottle. Dilution to a smaller volume may be needed for specific client requests. If only one graduated cylinder is to be used to measure all samples, then bring the blank to volume after the rest of the samples. This will help to determine if there is any sample carry over contamination.

Note: If a sample is allowed to go dry at any stage of the digestion procedure, the sample must be discarded and redigested.

7.6. If silicates or other insoluble material that could clog the nebulizer are present in the samples, allow the samples to settle overnight prior to analysis. If there is concern that the insoluble material will not settle out of solution, filter the sample with Whatman No. 41 filter paper. Note on digest log if samples had to be filtered (under comments).

7.7. In the digestion log record the start date/time, finish date/time, and the date/time digestates are relinquished. Transfer the samples to the metals analysis department. Have the analyst accepting the digestates initial and date the digestion log.

8. Quality Control

8.1. In each batch or SDG consisting of no more than 20 samples, a preparation blank is created. This consists of an empty, acid-washed Erlenmeyer flask to which the appropriate reagents are added and digested in exactly the same manner as a sample. The blank should be labeled on the digestate bottle in the following way: B, date, instrument, S or W (for soil or water) and the sequence number of the digestion. Example: B091202ICPS01.

8.1.1. The method blank prepared along with the samples is used as a contamination check. Since the holding time for all analytes associated with this method have a holding time of 6 months, repreparation would not be an issue if contamination was traced to a specific reagent.

8.2. Spiked samples should be employed to determine accuracy. In each batch or SDG consisting of no more than 20 samples, either matrix spike/sample duplicate (MS/Dup) or matrix spike/matrix spike duplicate (MS/MSD) samples will be prepared. For SW846-specified quality control measures, MS/Dup samples will be prepared in the following way. Dispense one sample in triplicate. A sample is chosen at random from the samples to be prepared and analyzed as a duplicate and matrix spike. Two will be digested exactly as any other sample. The third must be spiked prior to any acid addition, with the appropriate spike solutions determined by the analyst, and then treated as any other sample. All glassware and digestate bottles must be marked appropriately. If MS/MSD samples are required instead of the SW846-specified MS/Dup, both the second and third aliquots must be spiked prior to acid addition and all glassware and digestate bottles marked appropriately. See Appendix C for

instructions on the preparation and use of QC solutions. Spiking levels presented are to be used only as guidance. The actual analytes and concentrations may vary.

8.3. Determine the analytes required for analysis by consulting sample work order information. Each spiking solution is given its own unique number according to the page and line of the standards logbook which it is entered into. This number and the volume dispensed must be clearly recorded on the digestion log.

8.4. In each batch or SDG consisting of no more than 20 samples, either a Blank Spike (BLK SPK) or a Laboratory Control Sample (ICP LCSS) must be digested in exactly the same manner as a sample. A Blank Spike consists of an empty, acid-washed Erlenmeyer flask (or digestion vessel) to which the appropriate spikes are followed by the addition of reagents (determined as outlined in Section 7.3). The Certified Value and Advisory range for each analyte in a particular LCSS can be accessed from the Quality Control Database.

9. References

9.1. USEPA, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, most recent version, Method 3050B.

Appendix A

Digestion Benchsheet and Log

LAUCKS TESTING LABORATORIES, INC.		ICP DIGESTION LOG	Place a √ in the box next to the method performed.			
	Digest start : date/time	Digest finish : date/time	SW-846 3005A		ILM04.0-water	
SDG #:			SW-846 3010A		ILM04.0-soil	
Analyst:			SW-846 3050A		SW-846 3050B	

	Sample ID	Sample Size (g:mL)	Final Volume (mL)	Color Bef	Color Aft	Clarity Bef	Clarity Aft	Text	Art	Comments	Bottle #
01											
02											
03											
04											
05											
06											
07											
08											
09											
10											
11											
12											
13											
14											
15											
16											
17											
18											
19											
20											
21											
22											
23											
24											

Chain of Custody	Digest Relinquished Date / Time : Analyst	Digest Accepted Analyst	Digest Disposed Date	Hot Plate Temperature, °C
Reagents Lot Number	HNO_3	HCl	H_2O_2	

Color 1=Red, 2=Blue, 3=Yellow, 4=Green, 5=Orange, 6=Violet, 7=White, 8=Brown, 9=Grey, 10=Black, 11=Colorless
Clarity 1=Clear, 2=Cloudy, 3=Opaque
Texture F=Fine (powdery), M=Medium (sandy), C=Coarse (rocky)
P:\metals\bnchshee\icpdig.doc
Artifacts: If "Yes", give description in the comments field.

P:\metals\bnchshee\icpdig.doc **Spike solutions:** **spike witness:**

Appendix B

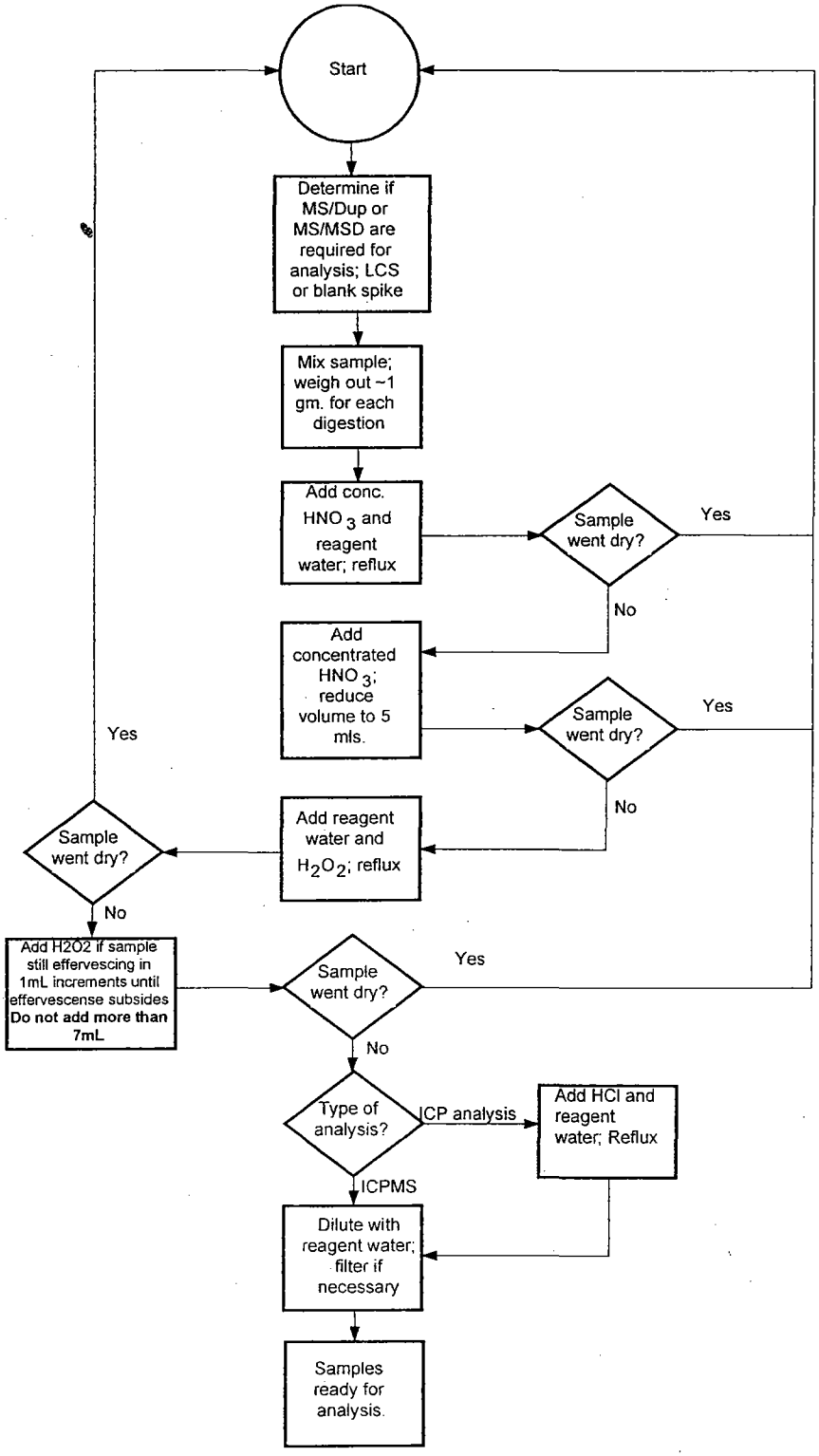
Checklist for 3050B

SW846-3050B

1.0-1.5g Sample
Add Spikes
5 mL H_2O
5 mL HNO_3
Heat 15 min
Cool
5 mL HNO_3
Heat 30 min
Repeat (5 mL HNO_3) until no brown fumes exist
Reduce to 5 mL or heat 2 hours
Cool
2 mL H_2O
3 mL H_2O_2
Heat 10 min. or until effervescence subsides
Cool
Repeat if effervescence continues with 1 mL aliquots H_2O_2 until effervescence is minimal or 7 times
Reduce to 5 mL or heat 2 hours
Cool
10 mL HCl (Omit for ICPMS)
Heat 15 min
Cool
Final Volume 100 mL

Appendix C

Flowchart


Start
Determine if MS/Dup or MS/MSD are required for analysis; LCS or blank spike
Mix sample; weigh out ~1 gm. for each digestion
Add conc. HNO 3 and reagent water; reflux
Sample went dry?
Yes
No
Add concentrated HNO 3; reduce volume to 5 mls.
Sample went dry?
Yes
No
Add reagent water and H2O2; reflux
Sample went dry?
Yes
No
Add H2O2 if sample still effervescing in 1mL increments until effervescense subsides Do not add more than 7mL
Sample went dry?
Yes
No
Type of analysis?
ICP analysis
Add HCl and reagent water; Reflux
ICPMS
Dilute with reagent water; filter if necessary
Samples ready for analysis.

Appendix D

Quality Control Solutions

ICP Spikes

- Prior to acid addition, the blank spike (unless the ICP LCSS is used) and the matrix spike are spiked with 1000 µLs of CLP spike solution and 5 mL of LTL-6 spike solution.

- The CLP spike solution for ICP digest was made by diluting 10 mLs of CLPP-SPK-1™, 10 mLs of CLPP-SPK-2™, and 10 mLs of CLPP-SPK-3™ to 100 mLs with DIW.

- The LTL-6 spike solution for ICP digest is made by diluting 50 mL 10000 ppm Ca solution, 50 mL 10000 ppm K solution, 50 mL 10000 ppm Na solution, and 50 mL 10000 ppm Mg solution (purchased from Inorganic Ventures) to 500 mL with DIW.

Analytes not present in the CLP or LTL-6 spiking solutions are added individually from vendor supplied 1000 ppm standards. Samples are spiked prior to digestion with 1000 µLs of these standards. The concentration of these analytes would be 1000 ppb in the digestate with a final volume of 100 mL.

ICP Analytes

Analyte	Concentration in Digest (ppb)	Analyte	Concentration in Digest (ppb)
Ag	50	Fe	1000
Al	2000	Mn	500
As	2000	Ni	500
Ba	2000	Pb	500
Be	50	Se	2000
Cd	50	Sb	500
Co	500	Tl	2000
Cr	200	V	500
Cu	250	Zn	500
Mg	50000	Ca	50000
Na	50000	K	50000

Table based on a final volume of 100 mL.

ICP-MS Spikes

- The ICPMS spike solution for the ICP-MS digest is vendor purchased and contains various routine analytes (currently supplied by SPEX).
- Add 5 mLs of ICPMS spike solution to samples prior to the addition of nitric acid

ICP-MS Analytes

Analyte	Concentration in Digest at instrument (ppb)	Analyte	Concentration in Digest at instrument (ppb)
Ag	50	Fe	2500
Al	2500	Mn	50
As	50	Ni	50
Ba	50	Pb	50
Be	50	Se	50
Cd	50	Sb	50
Co	50	Tl	50
Cr	50	V	50
Cu	50	Zn	50
Mg	5000	Ca	5000
Na	5000	K	5000

Table based on a final volume of 100 mL and a 5X dilution at the instrument.

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #: LTL-7202

Title: Metals Analysis Using Inductively Coupled Plasma - Mass Spectrometry (ICP/MS), SW 846 Method 6020

Revision history:

Number	Date
1.0	04/26/95
2.0	02/09/98
3.0	4/22/98
4.0	7/22/99
5.0	11/11/02
6.0	09/27/04
7.0	09/26/05

Revised by: Bill Ambacher Date: 9/28/05
Bill Ambacher, Inorganics Manager

Approved by: Harry Romberg Date: 9/29/05
Harry Romberg, QA Officer

Approved by: Kathy Kreps Date: 9/30/05
Kathy Kreps, Laboratory Director

Controlled Document

No. 20 **Assigned to:** TETRA

Table of Contents

1. INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
1.2 PERSONNEL QUALIFICATIONS 4
1.3 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 4
1.4 DEFINITION OF TERMS 4

2. EQUIPMENT LIST AND STANDARDS 7

2.1 INSTRUMENTATION: 7
2.2 STANDARDS 7
2.3 INTERNAL STANDARDS 7

3. SAFETY PRECAUTIONS AND WASTE DISPOSAL 8

3.1 SAFETY PRECAUTIONS 8
3.2 WASTE DISPOSAL 8

4. QUALITY CONTROL 9

4.1 METHOD DETECTION LIMIT STUDY 9
4.2 LINEAR RANGE STUDY 9
4.3 INTERNAL STANDARDS 9
4.4 INITIAL CALIBRATION 10
4.5 INITIAL CALIBRATION VERIFICATION 10
4.6 INITIAL CALIBRATION BLANK 11
4.7 CONTINUING CALIBRATION VERIFICATION (CCV) AND CONTINUING CALIBRATION BLANK (CCB) 11
4.8 INTERFERENCE CHECK SOLUTIONS A (ICSA) AND AB (ICSAB) 12
4.9 METHOD BLANKS 13
4.10 LABORATORY CONTROL SAMPLE 14
4.11 MATRIX SPIKE 14
4.12 POST-DIGESTION SPIKE 15
4.13 MATRIX SPIKE DUPLICATE/SAMPLE DUPLICATE 16
4.14 SERIAL DILUTION 17

5. PROCEDURE 17

5.1 INSTRUMENTAL CONDITIONS 17
1.2 INTERFERENCE PROBLEMS 19

6. TROUBLESHOOTING 20

6.1 TROUBLESHOOTING 20

7. REPORTS 21

7.1 DATA PACKET ORGANIZATION 21
7.2 REFERENCES: 21

APPENDIX I 23

STANDARD SOLUTIONS, ICV SOLUTION AND INTERNAL STANDARD SOLUTIONS. 23

APPENDIX II 27

CRI, ICSA AND ICSAB SOLUTIONS 27

APPENDIX III **29**
QC SUMMARY TABLE 29
APPENDIX IV **32**
AGENCY CRITERIA 32
APPENDIX V **36**
FLOW CHART 36
APPENDIX VI **38**
ROUTINE REPORTING LIMITS 38
APPENDIX VII **39**
MULTIPLE MASS MONITORING 39
APPENDIX VIII **40**
COMMON MOLECULAR ION INTERFERENCES IN ICP-MS 40
COMMON MOLECULAR ION INTERFERENCES IN ICP-MS (CONT'D) 41

1. Introduction and Scope

1.1 Method Description

1.1.1 Inductively coupled plasma-mass spectrometry (ICP-MS) is a technique which is applicable to sub-parts per billion concentrations of a large number of elements in water and wastes after appropriate sample preparation steps are taken. When dissolved constituents are required, samples must be filtered and acidified prior to analysis. No further digestion is required prior to analysis for dissolved elements. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soil, sludge, sediment, and other solid waste for which total (acid-leachable) elements are required.

1.1.2 See Appendix I for analytical masses and standard concentrations.

1.1.3 See Appendix VI for reporting limits.

1.1.4 See Appendix VII for additional masses that should be monitored.

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 their ability to perform the described analysis.

1.3 Sample Collection, Sample Storage, Holding Times

1.3.1 Water samples should to be collected in plastic or Teflon containers and preserved to a pH<2. A one liter sample bottle is sufficient volume for analysis. Soil samples do not require preservation (although we generally refrigerate at 4±2°C) and may be collected in glass if plastic containers are not available. At least 200 grams of sample should be collected. The holding time for metals is 6 months. If mercury is being analyzed by this technique, the holding time is 28 days.

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.

CCB - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis

sequence to determine whether the instrument or system has maintained a stable baseline.

CCV - Continuing calibration verification - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

CLP - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

DIW - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

ICB - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same matrix as calibration standards, without target analytes.

ICV - Initial calibration verification - This term is borrowed from the CLP protocol. It is a standard which is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate.

IDL - Instrument detection limit. IDL's can be estimated by analyzing seven replicates of a standard analyte solution over three nonconsecutive days. The analyte concentration should be 3-5 times the estimated IDL. Multiplying the average standard deviation by three will yield the IDL for that analyte. Each measurement must be performed as though it were a separate analytical sample. IDL's must be determined quarterly

MDL - Method detection limit - The lowest concentration of an analyte which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined and are performed annually.

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.

LRCS – Linear Range Check Standard- A standard prepared so that the concentrations of the target analytes are at a predetermined linear range. This standard is used to verify that the instrument or system is capable of reaching the predetermined values of the target analytes on an ongoing basis.

Serial Dilution - If the analyte concentration is within the linear range of the instrument and is sufficiently high (minimally, a factor of 100 above the IDL/MDL), an analysis of a fivefold dilution must agree within ±10% of the original determination. If not, an interference effect must be suspected. One serial dilution must be analyzed for each twenty samples or less of each matrix in a batch. A serial dilution is denoted in the raw data by an "L".

CRI – CRDL Check Standard - A sample of acidified DIW to which a known amount of elements is added. This is at a low concentration to assess the validity of the curve near the reporting limit.

ICSA - Interference Check Solution A - The ICSA is a solution that contains the interfering analytes. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the analytes of interest.

ICSAB - Interference Check Solution AB - The ICSAB is a solution that contains the interfering analytes and the analytes of interest. This solution is analyzed to indicate if a high level of interfering compounds will have an affect on the recovery of the analytes of interest.

Internal Standards - Internal standards are added to all blanks, standards, and samples. They monitor the affect of a sample's matrix on the quantification of the analytes of interest. The internal standards used for the Agilent 7500 are Li6, Sc45, Ge70, Ge72, Ge74, Y89, In115, Tb159, and Bi209. The internal standards used for the Elan 6100 are Li6, Sc45, Ge72, In115, Tb159 and Bi209.

Post-Digestion Spike - An analyte spike added to a portion of a prepared sample should be recovered to within 75% to 125% of the known value or within the laboratory derived acceptance criteria.

Standard-Addition - The standard addition technique involves adding known amounts of standard to an aliquot of the sample. This technique compensates for a sample constituent that enhances or depresses the analyte signal thus producing a different slope than that of the calibration standards.

QC Period - Quality control period - An analysis sequence initiated by the analysis of one or more standards, followed by samples, and terminated with a standard and blank

analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP

RSD or %RSD - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

2. Equipment List and Standards

2.1 Instrumentation:

2.1.1 Perkin-Elmer ELAN 6100 ICP-MS

2.1.1.1 Elan Software Version 2.31

2.1.1.2 Pentium Computer (or equivalent)

2.1.1.3 Perkin-Elmer AS-91 Autosampler or AS-90 Autosampler.

2.1.2 Agilent 7500C ICP-MS

2.1.2.1 ICP-MS ChemStation Revision B.02.04

2.1.2.2 Pentium Computer (or equivalent)

2.1.2.3 ASX-510 Autosampler

2.1.3 High purity argon, hydrogen, and helium

2.2 Standards

2.2.1 SW 846 requires the use of at least two standards and a blank but the PE Elan 6100 and the Agilent 7500C use five standards and a blank. Standards are matrix matched to the acid composition of the samples after digestion or preservation.

2.2.2 See Appendix I for standard concentrations.

2.3 Internal Standards

2.3.1 See Appendix I for standard concentrations.

3. Safety precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Routine precautions

3.1.2.1 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.2.2 Routine precautions include an awareness of the moving parts on the instrument you are using. These parts are often charged with power from an electrical component or with high pressure gas and have the potential to do harm if not used properly.

3.1.3 Electrical shock

3.1.3.1 All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.4 Radio Frequency

3.1.4.1 The ELAN 6100 and the Agilent 7500C use a high energy RF generator. Although the generators are well shielded, care should be taken when operating the instrument. Pace makers can be adversely affected by exposure to high energy RF.

3.1.5 Plasma Radiation

3.1.5.1 Do not view the torch without the proper eyewear. Severe eye damage can occur if the plasma is viewed directly.

3.2 Waste Disposal

3.2.1.1 Waste is collected at the instruments and neutralized prior to disposal down a sink. The amount of neutralized waste is recorded in the disposal log.

3.2.1.2 Digestions are stored for up to 6 months and then neutralized prior to disposal. The amount of neutralized waste is recorded in the disposal log.

3.2.1.3 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on waste segregation and disposal.

4. 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. Lauck's estimated MDLs are found in Appendix VI. Current MDL values are found in the iLIMS MDL database and associated raw data are stored in department files.

4.2 Linear range study

4.2.1 Method 6020 does not require a linear range study.

4.2.2 DOD requirements specify that every six months, linear ranges for each analyte are determined by analyzing a high concentration "sample". The analytically determined concentration must be within 10% of the true value. The true value is the upper limit of the ICP/MS linear range.

4.3 Internal Standards

4.3.1 PE 6100: A 50 µL aliquot of a 2 ppm of Sc45, 2 ppm of Ge74, 1 ppm of In115, 1 ppm of Li6, 1 ppm of Tb159 and 1 ppm of Bi209 is added to a 10 mL aliquot of all standards and samples prior to analysis.

4.3.2 Agilent: The internal standards are added online using a 2 ppm solution of Li6, a 2.5 ppm solution of Ge72, and a 1 ppm solution of Y89, In115, Tb159, and Bi209. This gives an effective concentration of 100, 125, and 50 ppb respectively in all standards and samples.

4.3.3 Instrument Criteria

4.3.3.1 The intensities of all internal standards for instrument check standards must be between 80 and 120 percent of the intensities of the internal standards in the initial calibration standard.

4.3.4 Corrective action.

4.3.4.1 If the criteria are not met, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and re-analyze the affected samples.

4.3.5 Sample Criteria

4.3.5.1 The intensities of all internal standards in the samples must be between 30 and 120 percent of the intensities of the internal standards in the initial calibration standard.

4.3.6 Corrective action

4.3.6.1 When the intensity of any internal standard in the sample fails to fall between the required levels, the sample must be diluted fivefold (1+4) and re-analyzed with the addition of appropriate amounts of internal standards. This procedure must be repeated until all internal standards fall within the prescribed windows.

4.4 Initial Calibration

4.4.1 Analyze standard solutions using a minimum of a calibration blank and three standards.

4.4.2 Criteria:

4.4.2.1 Initial calibration data is evaluated using the correlation coefficient of a linear regression analysis. The correlation coefficient must be 0.995 or greater. All CCVs and sample extract concentrations must be computed using the regression equation.

4.4.3 Corrective Action:

4.4.3.1 If the calibration does not meet this criterion, then the system must be recalibrated.

4.5 Initial Calibration Verification

4.5.1 Criteria

4.5.1.1 Immediately after the calibration curve, analyze a standard from a source other than that from which the calibration material was obtained. The calculated concentration of the ICV should be within 90%-110% of the true value.

4.5.2 Corrective action.

4.5.2.1 If the ICV criteria are not met, the analysis is terminated. Perform system maintenance and re-calibrate the instrument.

4.6 Initial Calibration Blank

4.6.1 Criteria

4.6.1.1 After the analysis of the ICV standard an instrument blank (ICB) is analyzed. The levels of target analytes in the ICB should be less than or equal 1/2 of the reporting limit. (See Appendix VI for reporting limits.)

4.6.2 Corrective action

4.6.2.1 If the initial ICB contains target analyte levels above 1/2 of the reporting limit, the system is out of control. The source of contamination must be identified and corrected before proceeding with the analysis.

4.7 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB)

4.7.1 A continuing calibration verification standard is analyzed after every 10 samples. Immediately following the CCV, a blank (CCB) solution is analyzed. In addition, this standard and blank must be the last samples analyzed in the run. Periodically, the CCV must be analyzed at a different level within the calibration range than routinely analyzed. A copy of this CCV must be available for review.

4.7.2 CCV

4.7.2.1 Criteria

4.7.2.1.1 The CCV must fall within ± 10% of the true value.

4.7.2.2 Corrective action

4.7.2.2.1 If CCV limits are exceeded, check calculations or perform instrument maintenance. Recalibrate and reanalyze. No sample results may be reported that are not bracketed by a successful calibration and a CCV which is in control or by preceding and following CCVs which are within limits.

4.7.3 CCB

4.7.3.1 Criteria

4.7.3.1.1 The levels of target analytes in the CCB should be less than or equal 1/2 of the reporting limit. (See Appendix VI for reporting limits.)

4.7.3.2 Corrective action

4.7.3.2.1 If the initial CCB contains target analyte levels above 1/2 of the reporting limit, the system is out of control. The source of contamination must be identified and corrected and the affected samples re-analyzed. As with the CCVs, no sample results may be reported that are not bracketed by a successful initial and continuing calibration blank which are in control or by preceding and following continuing calibration blanks.

4.8 Interference Check Solutions A (ICSA) and AB (ICSAB)

4.8.1 Due to the high sensitivity of the ICP-MS technique and instrument developments that have occurred since the method was written, the high dissolved solids content of the specified ICS solutions at normal concentrations are not recommended by the manufacturer for modern instruments. Laucks uses a 1:20 dilution of ICSA solution instead of method recommended levels.

4.8.2 See Appendix II for ICSA and ICSAB solutions concentrations.

4.8.3 ICSA:

4.8.3.1 Criteria

4.8.3.1.1 At the beginning of each run, an interference check solution A is analyzed. This solution contains interfering elements only. No other elements are present in the solution. All elements present must recover within 80-120% of true value. All elements not present should show a recovery within ± the contract required detection limit.

4.8.3.2 Corrective Action:

4.8.3.2.1 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.

4.8.4 ICSAB:

4.8.4.1 Criteria

4.8.4.1.1 At the beginning of each analytical sequence an ICSAB must be analyzed. This solution contains interfering elements and those elements that are interfered with.

(Refer to Appendix II for analytes present.) Analytes must recover between 80-120% of true value.

4.8.4.2 Corrective Action:

4.8.4.2.1 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 analysis.

4.9 Method Blanks

4.9.1 Criteria

4.9.1.1 Method blanks are used to verify contamination free reagents and apparatus. They are prepared with every set of samples prepared at the same time or at least one blank every 20 samples, whichever is more frequent. 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) 1/2 the reporting limit, or
(2) Five percent of the regulatory limit for that analyte, or
(3) Five percent of the measured concentration in the sample.

4.9.2 Corrective Action:

4.9.2.1 Corrective action may necessitate re-preparation and re-analysis of the sample set. For example if an analyte were found in the blank but not in any of the associated samples then sample group may not require re-analysis. In 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.

4.10 Laboratory Control Sample

4.10.1 The LCS is of the same matrix (soil or water) as the samples and is carried through the entire digestion procedure. Routinely, a blank spike is used for soil digestion and follows the same criteria as an LCSW. A soil LCS will be used only if the method specifies or upon client request. An LCS or blank spike analysis is performed with each digestion batch.

4.10.2 Criteria

4.10.2.1 At a minimum, LCSW (water) and soil blank spike control limits are 80% to 120%. LCSS (soil) control limits are supplied by the manufacturer. LCSS control limits are not derived by the laboratory due to the small number of data points available from each lot of certified material.

4.10.3 Corrective Action

4.10.3.1 If the LCS is not within the required control limits, then the digest 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 spikes and project requirements. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.11 Matrix Spike

4.11.1 A sample is chosen at random from the samples to be analyzed and an aliquot of spiking solution is added to this sample prior to preparation. Additional samples may be required by specific client requirements. The analyst should attempt to avoid selecting samples which are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. The minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{Recovery},\% = \frac{(SS - S)}{SA} * 100$$

where:
SS = concentration in spiked sample
S = native concentration in unspiked sample

SA = spike added, the amount of spiking material actually added calculated on the sample basis.

4.11.2 Criteria

4.11.2.1 For ICP/MS, control limits for spike recoveries are 75-125%, unless the sample result is greater than 4 times the spike concentration, or unless otherwise specified in the project specific QAPP. In-house control limits are based on historical performance and will change from time to time.

4.11.3 Corrective Action:

4.11.3.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of matrix spiking compounds from the SRM and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken. Data reported in validatable packages will be flagged with an "N" indicating the out-of-control event.

4.12 Post-Digestion Spike

4.12.1 Criteria

4.12.1.1 A post digestion spike is also performed on a portion of a prepared sample. The minimum frequency for PS analysis is 1 each per 20 samples per matrix. Control limits for post-spike recoveries will be 75-125%. Method 6020 requires analysis of a post-digestion spike.

4.12.2 Corrective Action:

4.12.2.1 When the post spike is recovered outside of the control limits, an explanation of the event should be included in the narrative.

4.12.2.2 When a matrix spike and a post spike are recovered outside the control limits of 75%-125% recovery, then the analyst will first look at Blank Spike recoveries and possibly reanalyze the matrix spike and post-spike. When necessary the analyst will dilute and reanalyze the sample, matrix spike and post spike to minimize the amount of interference in the sample.

4.12.2.3 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.

4.13 Matrix Spike Duplicate/Sample Duplicate

4.13.1 Method QC consists of MS/MSD. A duplicate may be performed instead of a MSD. Other types of QC can performed at the client's request. Method 6020 does not require analysis of MS or MSD, but does require a duplicate analyzed at a rate of 1 in 20. If MS/MSD is run per client requirement, the MSD can take the place of a matrix duplicate.

4.13.2 Criteria

4.13.2.1 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

4.13.2.2 For sample concentrations greater than 5 times the CRDL, control limits for RPD of duplicates will be ±20% unless otherwise specified in the project specific QAPP. For sample concentrations less than 5 times the CRDL, control limits for the difference will be ± the CRDL. In-house control limits are based on historical performance. The RPD control limits are detailed in the current QC Database and will change from time to time.

4.13.3 Corrective Action

4.13.3.1 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 RPD will be flagged with an "*".

4.14 Serial Dilution

4.14.1 Criteria

4.14.1.1 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%. If the difference is greater than 10% on analytes that exhibit a level 50 times greater than the IDL, then results for those analytes will be flagged with an "E" for validatable data packages.

4.14.2 Corrective Action:

4.14.2.1 No corrective is necessary other than appropriately flagging the data.

5. Procedure

5.1 Instrumental Conditions

5.1.1 Warm-up

5.1.1.1 Refer to manufacturer's instruction and the instrument operational SOPs for specific operating procedures. Allow at least 45 minutes for the instrument to stabilize before initiating any analysis.

5.1.2 Instrument Daily Check

5.1.2.1 Refer to manufacturer's instruction and the instrument operational SOPs for specific instructions on performing a daily check.

5.1.3 Analytical Operation.

5.1.3.1 Calibrate the instrument using a calibration blank and 5 standards. Refer to Appendix I for the concentration levels.

5.1.3.2 All masses which could affect data quality are monitored to determine potential effects from matrix components on the analytes of interest. (See Appendix VII for additional masses that should be monitored.)

5.1.3.3 After the calibration has been established, an ICV solution, prepared from a separate source than the calibration curve, is analyzed to verify the validity of the curve. Measurements for the analytes of interest must be at ±10% of the true

value. A re-calibration and re-analysis is required for any analyte which falls outside the control limit.

5.1.3.4 Immediately following the ICV, an ICB is analyzed. Measurements for the analytes of interest must be less than ½ the reporting limit. (See Appendix VI for reporting limits.) A re-calibration and re-analysis is required for any analyte which falls outside the control limit.

5.1.3.5 Analyze the interference check samples (ICSA and ICSAB) prior to the analysis of samples

5.1.3.6 Analyze a CCV and a CCB once every 10 analytical samples and at the end of the run.

5.1.3.7 Dilute samples that exceed the established calibration range of the instrument.

5.1.4 Analytical Sequence:

5.1.4.1 PE ELAN 6100: 50µL of Internal Standard is added to 10 mL of all samples prior to analysis.

5.1.4.2 Agilent 7500C: Internal standards are added online.

S0	Blank
S1	Standard 1
S2	Standard 2
S3	Standard 3
S4	Standard 4
S5	Standard 5
ICV	Initial Calibration Verification
ICB	Initial Calibration Blank
CRI1	Low level check
LRCS1	Linear Range Check Standard
ICSA1	Interference Check Standard A
ICSAB1	Interference Check Standard AB
CCV1	Continuing Calibration Verification
CCB1	Continuing Calibration Blank
10 Samples	Analytical Samples
CCV2	Continuing Calibration Verification
CCB2	Continuing Calibration Blank
10 Samples	Analytical Samples
CCV3	Continuing Calibration Verification
CCB3	Continuing Calibration Blank

5.2 Interference Problems.

5.2.1 An interference is anything that causes the signal from an analyte to be different from the same signal for the same concentration of that analyte in a calibration solution.

5.2.2 Matrix Interferences

5.2.2.1 Interferences associated with sample introduction using a nebulizer/spray chamber system are common in ICP-MS determinations. These interferences are generally the result of either sample matrix effects that influence aerosol formation or the formation of ions in the plasma (surface tension, viscosity). Complex sample matrices may interfere with focusing ions into the sample and skimmer cone orifices, and focusing and transporting ions through the ion lenses and quadrupole.

5.2.3 Spectral Interferences

5.2.3.1 Spectral interferences are the result of other chemical species (isotopes or ions) which are present at the same atomic mass as the analyte of interest. Spectral interferences generally occur from isobaric overlaps, plasma-induced polyatomic ions, matrix solvent-induced polyatomic ions, and matrix-induced polyatomic ions.

5.2.3.2 See Appendix VIII for examples of common spectral interferences.

5.2.4 Isobaric Overlaps

5.2.4.1 Since most elements have more than one isotope, it is possible for the mass spectrum of an isotope of one element to directly overlap that of another element. Minor overlaps are corrected by measuring the intensity of the interfering element of major isotopes. The software of both instruments uses built-in calculation routines to automatically correct for known isobaric overlaps when calculating analyte concentrations in samples. In general, it is not necessary to make any further adjustments to the system.

5.2.4.2 For example, Vanadium needs to be corrected for isobaric interference from Chromium. The correction equation is:

$$V = [1.0 * (V^{51})] - 3.127 * [(Cr^{53})-(0.113)(Cr^{52})]$$

Note: the correction equations can be different depending on the instrument and technology.

5.2.5 Plasma-Induced Polyatomic Ions

5.2.5.1 The presence of atmospheric gases or the argon carrier is sources of potential interfering ions as the result of reaction with other analyte or matrix components. These interfering ions occur as polyatomic, molecular ions.

5.2.5.2 See Appendix VIII for examples of plasma-induced polyatomic ion interferences.

5.2.6 Matrix Solvent-Induced Polyatomic Ions.

5.2.6.1 The solvent or acid in which the sample is dissolved can also be a source of interfering polyatomic ions. Chlorine from hydrochloric or perchloric acid or salt water and sulfur from sulfuric acid all form polyatomic ions with argon and other plasma gases. For organic solvents, carbon and oxygen can form polyatomic ions. The most severe solvent-induced interference is from CO on silicon. All three silicon isotopes are overlapped by carbon species, precluding the determination of silicon in organic matrices.

5.2.6.2 See Appendix VIII for examples of matrix solvent-induced polyatomic ion interferences.

5.2.7 Matrix-Induced Polyatomic Ions

5.2.7.1 Polyatomic ions may also be generated by sample constituents, usually in combination with oxygen. Oxides of some heavier elements can be formed in the plasma/spectrometer interface. Typically, oxides are formed in relatively low concentration. In sufficient concentrations, oxide forming elements can interfere with determinations of analytes at higher mass. One example is the formation of molybdenum oxide which overlaps all major isotopes of cadmium.

5.2.7.2 See Appendix VIII for examples of matrix-induced polyatomic ion interferences.

5.2.8 Refer to the instrument manufacturer's manual or the instrument operational SOPs for how to identify these problems and procedures for correcting these effects.

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 SOP metals validation for a checklist detailing data packet organization.

7.1.2 If requested, data for all analyses performed under SW 846 guidelines can be reported via CLP forms.

7.1.3 Data packages will be produced via MARRS. Analyte levels that are less than the IDL will be reported as the SDL followed by a "U". Analyte levels that fall between the IDL 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.

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
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 my be required but are specified in project specific documents.

7.2 References:

Test Methods for Evaluating Solid Waste, SW-846, Method 6020, Revision 0, September 1994

Department Of Defense Quality Systems Manual for Environmental Laboratories, Version 3, March 2005

Air Force Center for Environmental Excellence (AFCEE) Quality Assurance Project Plan, Version 4.0.01, May 2005

Agilent 7500 ICP-MS ChemStation Operator's Manual, Agilent Technologies, Jan. 2001

Agilent 7500 ICP-MS Hardware Manual, Agilent Technologies, Jan. 2001

Agilent 7500 Series ICP-MS Installation Guide, Agilent Technologies, Feb. 2001

Agilent 7500 ICP-MS Series Integrated Sample Introduction System Manual, Agilent Technologies, June 2000

Elan 6000/6100 Software Guide 017236 Rev. B, Perkin Elmer, 1999

Elan 6100 Hardware Guide, Perkin Elmer, Dec. 1998

Elan 6000/6100 Software Upgrade Kit for Elan Software ver. 2.3.1, Perkin Elmer, Nov. 1999

USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, ILM05.3, Dec. 2001

Appendix I

Standard Solutions, ICV Solution and Internal Standard Solutions.

ELAN Calibration Solution

Elements	Mass	STD1, µg/L	STD2, µg/L	STD3, µg/L	STD4, µg/L	STD5, µg/L
Be	9	1	10	50	100	200
Na	23	100	1000	5000	10000	20000
Mg	25	100	1000	5000	10000	20000
Al	27	50	500	2500	5000	10000
K	39	100	1000	5000	10000	20000
Ca	44	100	1000	5000	10000	20000
V	51	1	10	50	100	200
Cr	52	1	10	50	100	200
Mn	55	1	10	50	100	200
Fe	57	50	500	2500	5000	10000
Co	59	1	10	50	100	200
Ni	60	1	10	50	100	200
Cu	63	1	10	50	100	200
Zn	66	1	10	50	100	200
As	75	1	10	50	100	200
Se	82	1	10	50	100	200
Ag	107	1	10	50	100	200
Cd	111	1	10	50	100	200
Sb	121	1	10	50	100	200
Ba	137	1	10	50	100	200
Tl	205	1	10	50	100	200
Pb	208	1	10	50	100	200
Li	7	1	10	50	100	200
B	11	1	10	50	100	200
Mo	98	1	10	50	100	200
Sn	118,120	1	10	50	100	200
U	238	2	20	100	200	400
Sr	88	1	10	50	100	200
Th	232	2	20	100	200	400

The STD1, STD2, STD3, STD4, and STD5 are made by diluting 10, 100, 500, 1000, and 2000µL of the ICPMS stock standard, 10, 25, 100, 250, 500µL of the Hg standard, and diluting 10,100, 500, 1000, and 2000µL of the Th standard to a 50 mL final volume.

250 µL of ELAN internal standard should be added to all standards and blanks with a final volume of 50mL.

Agilent Calibration Solutions

Elements	Mass	STD1, µg/L	STD2, µg/L	STD3, µg/L	STD4, µg/L	STD5, µg/L
Be	9	1	10	50	100	200
Na	23	100	1000	5000	10000	20000
Mg	24	100	1000	5000	10000	20000
Al	27	50	500	2500	5000	10000
K	39	100	1000	5000	10000	20000
Ca	40	100	1000	5000	10000	20000
V	51	1	10	50	100	200
Cr	52	1	10	50	100	200
Mn	55	1	10	50	100	200
Fe	56	50	500	2500	5000	10000
Co	59	1	10	50	100	200
Ni	60	1	10	50	100	200
Cu	63	1	10	50	100	200
Zn	66	1	10	50	100	200
As	75	1	10	50	100	200
Se	78	1	10	50	100	200
Ag	107	1	10	50	100	200
Cd	111	1	10	50	100	200
Sb	121	1	10	50	100	200
Ba	137	1	10	50	100	200
Tl	205	1	10	50	100	200
Pb	208	1	10	50	100	200
Li	7	1	10	50	100	200
B	11	1	10	50	100	200
Mo	95	1	10	50	100	200
Sn	118	1	10	50	100	200
U	238	2	20	100	200	400
Sr	88	1	10	50	100	200
Th	232	2	20	100	200	400
Ti	47	1	10	50	100	200

The STD1, STD2, STD3, STD4, and STD5 are made by diluting 10, 100, 500, 1000, and 2000µL of the ICPMS stock standard, 10, 25, 100, 250, 500µL of the Hg standard, and diluting 10,100, 500, 1000, and 2000µL of the Th standard to a 50 mL final volume.

STOCK STANDARD SOLUTIONS

The ICPMS stock standard is custom made and currently purchased from AccuStandard.

Concentration	Analyte
500mg/L	Ca, Na, Mg, K
250mg/L	Al Fe
10mg/L	U
5mg/L	Sn, Ag, B, As, Be, Ba, Cd, Cr, Co, Cu, Li, Mn, Mo, Ni, Sb, V, Zn, Pb, Sr, Se, Ti, Tl

THE ICV STANDARD STOCK SOLUTION

The ICP/MS ICV standard consists of the following solutions to 500mL of 2%HNO_3:

mL	Analyte
12.5 of 10000ppm solution	Al, Fe
25 of 10000ppm solution	Ca, Mg, Na, K
2.5 of 1000ppm solution	Sn, Ag, B, As, Be, Ba, Cd, Cr, Co, Cu, Li, Mn, Mo, Ni, Sb, V, Zn, Pb, Sr, Se, Ti, Tl
5.0 of 1000ppm solution	U

THE ICP/MS INTERNAL STANDARD SOLUTION

The ELAN internal standard consists of the following solutions to 200mL of 2%HNO_3:

1000 µL	1000 ppm Sc	500 µL	1000 ppm Bi
1000 µL	1000 ppm Ge	500 µL	1000 ppm Tb
500 µL	1000 ppm In	500 µL	1000 ppm Li^6

The Agilent internal standard consists of the following solutions to 200mL of 2%HNO_3:

20 µL	10000 ppm Y
400 µL	1000 ppm Li6
200 µL	1000 ppm In
500 µL	1000 ppm Ge
200 µL	1000 ppm Bi
200 µL	1000 ppm Tb
200 µL	1000 ppm Sc

Appendix II

CRI, ICSA and ICSAB Solutions

Element	Std CRI(PPB)	King County CRI (PPB)	ICSA (PPM)	ICSAB (PPM)
Aluminum	50	50	5	6
Antimony	1	1		0.020
Arsenic	1	1		0.020
Barium	1	1		0.020
Beryllium	1	1		0.020
Boron	5			0.020
Cadmium	1	2		0.020
Calcium	100	10	15	17
Chromium	1	5		0.020
Cobalt	1	3		0.020
Copper	2	2		0.020
Iron	50	5	12.5	13.5
Lead	1	1		0.020
Lithium	5			0.020
Magnesium	100	15	5	7
Manganese	5	1		0.020
Mercury	0.1			0.5
Molybdenum	1		0.10	0.12
Nickel	1	10		0.040
Potassium	100	300	5	7
Selenium	1	1		0.020
Silver	1	1		0.020
Sodium	100	500	12.5	14.5
Strontium	5			0.020
Thallium	1	1		0.020
Thorium	2			0.020
Tin	10	10		0.020
Titanium	5		0.10	0.12
Uranium	2			0.040
Vanadium	2	2		0.020
Zinc	10	4		0.020

The CRI stock standard consists of the following solutions to 100mL of 2%HNO3:

Be	20µL of 1000ppm
Na	100µL of 10000ppm
Mg	100µL of 10000ppm
Al	50µL of 10000ppm
K	100µL of 10000ppm
Ca	100µL of 10000ppm
V	40µL of 1000ppm
Cr	20µL of 1000ppm
Mn	100µL of 1000ppm
Fe	50µL of 10000ppm
Co	20µL of 1000ppm
Ni	20µL of 1000ppm
Cu	40µL of 1000ppm
Zn	200µL of 1000ppm
As	20µL of 1000ppm
Se	20µL of 1000ppm
Ag	20µL of 1000ppm
Cd	20µL of 1000ppm
Sb	20µL of 1000ppm
Ba	20µL of 1000ppm
Tl	20µL of 1000ppm
Pb	20µL of 1000ppm
Li	100µL of 1000ppm
B	100µL of 1000ppm
Mo	20µL of 1000ppm
Sn	200µL of 1000ppm
U	40µL of 1000ppm
Sr	100µL of 1000ppm
Th	100µL of 1000ppm
Ti	100µL of 1000ppm

The CRI is prepared by diluting 250µL of the CRI stock standard to a 50 mL final volume.

The ICSA Solution is currently purchased from CPI and contains:

3,000 ppm Ca	1,000 ppm Al, Mg, and K
2,500 ppm Fe and Na	20 ppm Mo and Ti

Appendix III

QC Summary Table

Laucks Testing Laboratories

Method SW-846 6020 QA Requirements and Corrective Actions

(See Appendix IV for Agency Specific Criteria)

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Mass Calibration Check	Must be performed in mass regions of interest and be within ±0.05 amu of the actual mass value.	Must be performed in mass regions of interest and be within ±0.05 amu of the actual mass value. See also operational SOPs	Beginning of each analysis.	Perform new mass calibration.	Instrument Logbook
Resolution Check	< 0.7 amu full width at 10% peak height	< 0.7 amu full width at 10% peak height	Beginning of each analysis.	Adjust resolution.	Instrument Logbook
Initial Calibration	Blank and at least one standard	Blank and at least three standards, correlation coefficient > 0.995	Beginning of each analysis	NA	In the raw data and/or on FORM 14.
Initial Calibration Verification	±10% of true value. Made from an independent source.	±10% of true value. Made from an independent source.	Immediately following calibration.	Recalibrate and reverify.	Form 2, in the raw data
Initial Calibration Blank	Values must be < 3x the IDL for each element.	Values must be < 1/2 CRDL	Immediately following ICV.	Recalibrate, reverify, and rerun the ICB.	Form3, in the raw data
Continuing Calibration Verification	±10% true value.	±10% true value. Analyte levels are at the mid-range of the calibration.	Every 10 samples and end of run.	Recalibrate and rerun affected samples.	Form 2, in the raw data
Continuing Calibration Blank	Values must be < 3 times the IDL for each element.	Values must be < 1/2 CRDL	Immediately following CCV.	Recalibrate and rerun affected samples.	Form 3, in the raw data
Method Blank	< CRDL or <5% of regulatory limit or any sample	< 1/2 CRDL or <5% of regulatory limit or any sample	One/batch	Redigest samples	Form 3, in the raw data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Internal Standards	1.Samples: 30 % - 120 % of the initial calibration standard 2. Instrument Check Standards: 80 -120 of the initial calibration standard.	1. Samples 30 % - 120 % of the initial calibration blank. 2. Instrument Check Standards: 80 -120 of the initial calibration blank.	1. All samples 2. All instrument check standards	1. Dilute and reanalyze. 2. Recalibrate, re-analyze the affected samples.	In the raw data.
Serial Dilution	Within ±10% of the original value if the analyte conc. is > 100 x the IDL.	Within ±10% of the original value if the analyte conc. is > 100 x the IDL.	One/batch	Flag data with an "E".	Form 9,.in the raw data
Duplicate, % Difference	± 20% for analyte values greater than 100 times the IDL.	± 20% or current QC database criteria for analyte values greater than 5x the CRDL.	5% or per batch	reanalyze digestates, if still fail, consult QCO	Form 6, in raw data, or database report
Matrix Spike Recovery	Not required by 6020.	75-125%, per client criteria, or current QC database criteria.	5% or per batch	Consult QCO	Form 5A, in raw data, or database report
Post-Digestion Spike	75%-125%	75%-125%	5% or per batch	dilute and re-analyze or MSA	Form 5B, in the raw data
LRCS (Linear Range Check Standard)	Not required by 6020.	90%-110%	Semi-annually	Report only to high calibration standard.	In the raw data.
Laboratory Control Sample	One/batch, no acceptance criteria	LCSW/BS: 80%-120% LCSS: Manufacturer Specs.	One/batch	Redigest samples.	Form 7, in raw data, or database report
ICSA and ICSAB	±20% true value of analytes, or ± the CRDL.	±20% true value of analytes, or ± the CRDL.	Beginning and end of run	reanalyze affected samples	Form 4, or in raw data.
CRI	Not required	No currently required limits.	Beginning of run	none	Form 2B, or in raw data.

Appendix IV

Agency Criteria

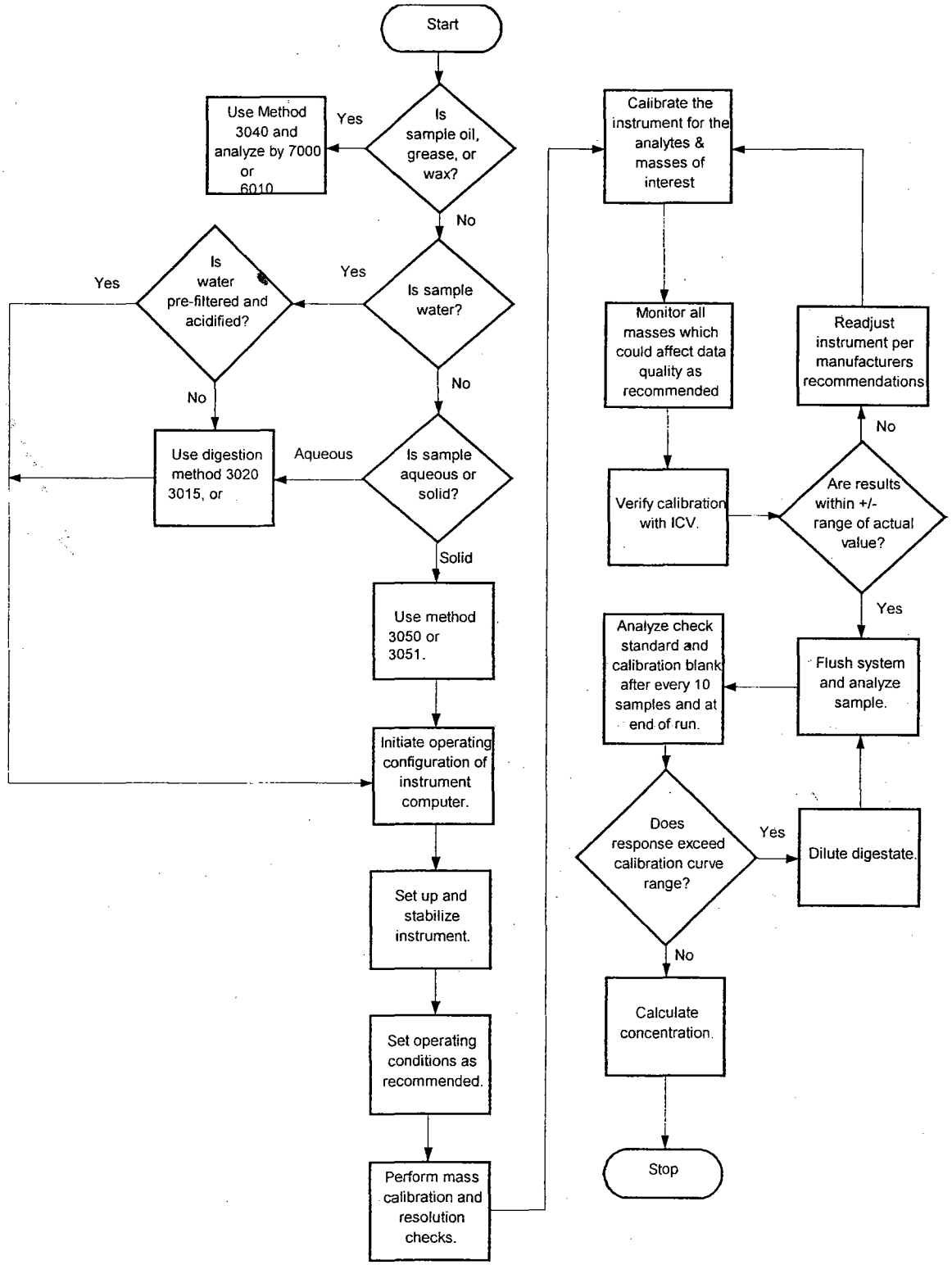
Study/QC	SW-846 6020		DOD-QSM (Ver. 3)		AFCEE (4.0)		ILM 4.1		ILM 5.3		200.8 (Rev. 5.5)	
	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency
IDLs	---	3 mo.	Detection limits <= MDL	3 mo.	Detection limits <=MDL.	Initially	N/A	N/A	---	---	---	---
MDLs	---	---	---	Initially & once per 12 mo.	---	Initially & once per 12 mo. or quarterly MDL verification checks	N/A	N/A	<1/2 CRQL	Initially & every 12 mo.	---	12 mo.
Linear Range Study	---	---	---	---	---	---	N/A	N/A	---	Initially	+/- 10%	Initially
Linear Range Check Std.	---	---	+/- 10%	6 mo.	+/- 10%	3 mo.	N/A	N/A	+/- 5%	3 mo.	+/- 10%	12 mo
Tuning	Mass calibration <=0.1 amu, resolution <0.9 amu full width at 10% peak height	Prior to initial calibration	Mass calibration <=0.1 amu, resolution <0.9 amu full width at 10% peak height	Prior to initial calibration	Mass calibration <=0.1 amu, resolution <0.9 amu full width at 10% peak height	Prior to initial calibration	N/A	N/A	Mass calibration <=0.1 amu	Prior to initial calibration	Mass calibration <=0.1 amu, resolution approx. 0.75 amu at 5% peak height	Prior to initial calibration
Internal Standards (IS)	IS intensity w/in 30-120% of intensity of the IS in the initial calibration	Every sample	IS intensity w/in 30-120% of intensity of the IS in the initial calibration	Every sample	IS intensity w/in 30-120% of intensity of the IS in the initial calibration	Every sample	N/A	N/A	60-125% of the original response in the calibration blank	Every sample	60-125% of the original response in the calibration blank	Every sample
ICV/CCV	+/- 10%	Immed. after calib.(ICV), prior to & after every 10 samples (CCV), and at 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)	N/A	N/A	+/- 10%	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)	ICV +/- 10%, CCV +/- 15% (after initial CCV)	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), after every 10 samples (CCB), and 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), after every 10 samples (CCB), and end of run (CCB)	N/A	N/A	<= CRQL	Immed. after calib.(ICB), after every 10 samples (CCB), and end of run (CCB)	<2.2xMDL (PB)	Immed. after calib., after every 10 samples, and end of run (ICB/CCB). One in 20 per batch per matrix (PB)

Study/QC	SW-846 6020		DOD-QSM (Ver. 3)		AFCEE (4.0)		ILM 4.1		ILM 5.3		200.8 (5.5)	
	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency
Low Level Check Std. (CRI/CRDL/CRQL)	None		+/- 20%	Daily after init. calib. For single pt. curve analysis	+/- 20%	Prior to analysis of samples. Not required if multipoint (3+) curve with low std <=RL	N/A	N/A	+/- 30%. (+/- 50% for Co, Mn, Zn)	Every 20 samples. Initial "CRI" at the CRQL immediately followed by ICSA/ICSAB. Subsequent CRIs are immediately followed by CCV/CCB. CRI counts as a sample.	None	---
ICSA	None	At beginning of analytical run or once every 12 hours	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 or once every 12 hours	N/A	N/A	+/- 3x CRQL or +/-20% of true value	At beginning of analytical run. Not greater than 20 samples per analysis run.	---	---
ICSAB	None	At beginning of analytical run or once every 12 hours	+/- 20%	At beginning of analytical run	+/- 20%	At beginning of analytical run or once every 12 hours	N/A	N/A	+/- 3x CRQL or +/-20% of true value	At beginning of analytical run. Not greater than 20 samples per analysis run.	---	---
LCS (BS)	None	One in 20 per prep batch	80-120% (per DoD-QSM Appendix DoD-D)	One per prep batch	80-120%	One per prep batch	N/A	N/A	H_2O: 80-120%. Soil: No criteria.	One per prep batch per matrix	85-115%	One per prep batch per matrix
Matrix Spike (MS)	None	---	Per DoD LCS limits	One per prep batch per matrix	H_2O & Soil: 80-120%.	One MS/MSD per 20 samples	N/A	N/A	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) or Matrix Spike Duplicate (MSD)	RPD <= 20% for sample levels >100x IDL	One in 20 per prep batch per matrix	RPD < 20%	One per prep batch per matrix	Matrix Spike:: H_2O: RPD <= 15%, Soil: <=25%	One MS/MSD per 20 samples (No Duplicate)	N/A	N/A	RPD <= 20% if analyte level >=5x CRQL. +/-CRQL, otherwise.	One per prep batch per matrix	---	---
Serial Dilution (L)	% Diff <= 10% (Only for sample concs. >100xMDL)	One in 20 per prep batch per matrix	% Diff <= 10% (Only for sample concs. >100xMDL)	One per prep batch	% Diff <= 10% (Only for sample concs. >=100 x MDL)	One per prep batch per matrix	N/A	N/A	% Diff <= 10%, if analyte conc. >50x MDL	One per prep batch per matrix	---	---

Study/QC	SW-846 6020		DOD-QSM (Ver. 3)		AFCEE (4.0)		ILM 4.1		ILM 5.3		200.8 (Rev. 5.5)	
	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency
Post Digestion Spike (PS)	75-125%	Not specified	75-125%, unless sample level >4x spike level	When dilution test fails or if an analyte's conc. for all samples in a batch is <100 x MDL	75-125%	When dilution test fails or if an analyte's conc. for all samples in a batch is <100 x MDL	N/A	N/A	75-125%, unless sample level >4x spike level	When the MS fails	---	---

Appendix V

Flow Chart


Start
Is sample oil, grease, or wax?
Yes
Use Method 3040 and analyze by 7000 or 6010
No
Is sample water?
Yes
Is water pre-filtered and acidified?
Yes
No
Use digestion method 3020 3015, or
Aqueous
Is sample aqueous or solid?
No
Solid
Use method 3050 or 3051.
Initiate operating configuration of instrument computer.
Set up and stabilize instrument.
Set operating conditions as recommended.
Perform mass calibration and resolution checks.
Calibrate the instrument for the analytes & masses of interest
Monitor all masses which could affect data quality as recommended
Verify calibration with ICV.
Are results within +/- range of actual value?
No
Readjust instrument per manufacturers recommendations
Yes
Flush system and analyze sample.
Analyze check standard and calibration blank after every 10 samples and at end of run.
Does response exceed calibration curve range?
Yes
Dilute digestate.
No
Calculate concentration.
Stop

Appendix VI

Routine Reporting Limits

SW 846 6020 Reporting Limits and MDLs

Elements	Mass	Estimated MDL, µg/L	Reporting Limit, µg/L
Be	9	0.10	1
Na	23	10.0	100
Mg	24,25	5.0	100
Al	27	20.0	50
K	39	10.0	100
Ca	40,44	10.0	100
V	51	0.05	2
Cr	52	0.05	1
Mn	55	0.10	5
Fe	56,57	5.0	50
Co	59	0.05	1
Ni	60	0.10	1
Cu	63	0.20	2
Zn	66	2.5	10
As	75	0.05	1
Se	78,82	0.10	1
Ag	107	0.05	1
Cd	111	0.05	1
Sb	121	0.05	1
Ba	137	0.20	1
Tl	205	0.05	1
Pb	208	0.10	1
Li	7	0.10	5
B	11	0.50	5
Mo	95,98	0.05	1
Sn	118	0.05	10
U	238	0.05	2
Sr	88	0.10	5
Ti	47	0.10	5
Th	232	0.05	5

Reporting limits are approximately 2-10 times the instrumental MDL. The MDL is based on samples prepared using SW 846 3010. Values actually reported may be less than the routine reporting limits but above our method detection limit.

Appendix VII

Multiple Mass Monitoring

Additional masses that should be routinely monitored.

Elan

Analyte	Mass
Cr	53
Ni	62
Cu	65
Zn	67, 68
Se	77
Mo	95, 97
Ag	109
Ru	99
Pd	105
Kr	83
Cd	106, 108, 114
Sb	123
Ba	135
Hg	199, 200, 201
Tl	203
Pb	206, 207

Agilent

Analyte	Mass
Cr	53
Ni	62
Cu	65
Zn	67, 68
Se	77, 82
Mo	97, 98
Ag	109
Ru	99
Pd	105
Kr	83
Cd	106, 108, 114
Sb	123
Ba	135
Hg	199, 200, 201
Tl	203
Pb	206, 207

Appendix VIII

Common Molecular Ion Interferences in ICP-MS

Molecular Ion	Mass	Method Elements/Internal Standards affected
NH^+	15	
OH^+	17	
OH_2^+	18	
C_2^+	24	
CN^+	26	
CO^+	28	
N_2^+	28	
N_2H^+	29	
NO^+	30	
NOH^+	31	
O_2^+	32	
O_2H^+	33	
$^{36}ArH^+$	37	
$^{38}ArH^+$	39	
$^{40}ArH^+$	41	
CO_2^+	44	Sc
CO_2H^+	45	Cr
ArC^+, ArO^+	52	Cr
ArN^+	54	Mn
$ArNH^+$	55	
ArO^+	56	
$ArOH^+$	57	Se
$^{40}Ar^{36}Ar^+$	76	Se
$^{40}Ar^+$	78	Se

Common Molecular Ion Interferences in ICP-MS (cont'd)

Molecular Ion	Mass	Method Elements/Internal Standards affected
Bromide[12]		
$^{81}BrH^+$	82	Se
$^{79}BrO^+$	95	Mo
$^{81}BrO^+$	97	Mo
$^{81}BrOH^+$	98	Mo
$Ar^{81}Br^+$	121	Sb
Chloride		
$^{35}ClO^+$	51	V
$^{35}ClOH^+$	52	Cr
$^{37}ClO^+$	53	Cr
$^{37}ClOH^+$	54	Cr
$Ar^{35}Cl^+$	75	As
$Ar^{37}Cl^+$	77	Se
Sulfate		
$^{32}SO^+$	48	
$^{32}SOH^+$	49	
$^{34}SO^+$	50	V, Cr
$^{34}SOH^+$	51	V
SO_2^+, S_2^+	64	Zn
$Ar^{32}S^+$	72	
$Ar^{35}S^+$	74	
Phosphate		
PO^+	47	
POH^+	48	
$PO2^+$	63	Cu
ArP^+	71	
Group I, II Metals		
$ArNa^+$	63	Cu
ArK^+	79	
$ArCa^+$	80	
Matrix Oxides		
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

LAUCKS TESTING LABORATORIES INC.

Seattle, Washington

SOP #: LTL-7501

Title: Operation of the PE FIMS 400 / PE FIMS 100 for the Analysis of Hg for EPA Methods SW-846 7470/7471, 245.1/245.5, and CLP ILM04.1/ILM05.3

Number	Date
4	09/27/05
3	10/10/02
2	04/23/01
1	02/04/98
0	02/14/96

Revised by: Jennifer Ancona, Chemist Date: 9/29/05

Reviewed by: Bill Ambacher, Inorganics Manager Date: 9/29/05

Approved by: Harry Romberg, QA Officer Date: 9/29/05

Approved by: Kathy Kreps, Laboratory Director Date: 9/29/05

Controlled Document

No. 20 Assigned to: TETRA

Table of Contents

1 INTRODUCTION AND SCOPE 4
1.1 METHOD DESCRIPTION: 4
1.2 PERSONNEL QUALIFICATIONS 4
1.3 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 4
1.4 INTERFERENCES 4
1.5 DEFINITION OF TERMS 5
2 EQUIPMENT, STANDARDS AND REAGENTS 7
2.1 EQUIPMENT 7
2.2 STANDARDS 7
2.3 REAGENTS: 9
3 SAFETY PRECAUTIONS AND WASTE DISPOSAL 10
3.1 SAFETY PRECAUTIONS 10
4 CALIBRATION AND QUALITY CONTROL 10
4.1 METHOD DETECTION LIMIT STUDY (MDL). 10
4.2 INITIAL MULTI-POINT CALIBRATION: 11
4.3 CALIBRATION CRITERIA 11
4.4 CONTINUING CALIBRATION: 12
4.5 MATRIX SPIKE 12
4.6 MATRIX SPIKE DUPLICATE 13
4.7 SAMPLE DUPLICATE 13
5 OPERATION PROCEDURES 14
5.1 MOD BLOCK – PLASTIC VIAL – WATER OR SOIL (STANDARD PREPARATION): 14
5.2 MOD BLOCK DIGESTION – WATER SAMPLES 15
5.3 MOD BLOCK DIGESTION – SOIL SAMPLES 16
5.4 ANALYSIS ON FIMS 400 OR FIMS 100 17
5.5 REPORTING RESULTS FROM THE FIMS 400 OR FIMS 100 18
5.6 EXPORTING RESULTS FROM THE FIMS 400 OR FIMS 100 19
6 TROUBLESHOOTING 19
6.1 TROUBLESHOOTING 19
7 REPORTS 19
7.1 DATA PACKET ORGANIZATION 19
7.2 DATA QUALIFYING FLAGS 20
8 REFERENCES 20
APPENDIX I - QC SUMMARY TABLE 21
APPENDIX II - AGENCY CRITERIA 23

APPENDIX III – SAMPLE DIGESTION LOG **26**

APPENDIX IV - EXAMPLE REPORT FORM **31**

1 Introduction and Scope

1.1 Method Description:

1.1.1 This SOP covers the cold vapor analysis of Hg using the Perkin-Elmer Flow Injection Mercury System, Model 400 (FIMS 400) or Model 100 (FIMS 100). Sample handling and preparation are as required by SW-846 methods 7470/7471 and 245.1/245.5. This SOP is also valid for the preparation and analysis of samples under CLP protocol when used in conjunction with the SOW ILM04.1/5.3. Where the requirements of these procedures differ, the more stringent requirements are used. The requirement of analyzing soil samples in triplicate under 7471 is not used except upon request.

1.1.2 After preparation and digestion to convert Hg to its ionic form, 0.5 mL of sample is introduced into a stream of carrier solution (3% HCl), mixed with a $SnCl_2$ suspension to reduce the Hg ion to its metallic form, which is then carried into an absorption tube through which passes light at a wavelength of 253.7 nm. The absorption is then measured.

1.1.3 Laucks Testing Laboratories has studied the accuracy in using the MOD Block Digestor in place of the water bath for sample digestion. Due to plastic vial volume limitations, 50 mL of sample is used. MDL studies have found results comparable to the water bath in all methods.

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 stored either in glass or plastic. The holding time is 28 days from collection. Soil samples are stored at 4° ±2° C. Water samples are stored at room temperature. Water samples are preserved by the addition of HNO_3 to a pH <2.

1.4 Interferences

1.4.1 This section covers interferences that may potentially occur with the FIMS associated with sample or standard preparation or gas or liquid flow through the system.

1.4.2 Foaming

1.4.2.1 If excess foaming results in liquid being transported into the quartz cell, the reagent stream should be stopped immediately, and the tubing and cell assemblies should be disassembled and cleaned.

1.4.3 Precipitates

1.4.3.1 All solutions used with the FIMS (samples, carrier solutions, and reducing agents) must be free of solid particles since they are transported through narrow tubing and valve openings. In addition, the formation of precipitates during the course of a reaction must be avoided. When such a case occurs, an appropriate digestion or separation method should be used to remove the offending component.

1.4.4 Contamination

1.4.4.1 The detection limits obtainable using the FIAS-System are very often a function of the degree of contamination in the samples and the reagents rather than of instrument capabilities. Contamination is the main source of error when working at the nanogram levels typical for FIMS analyses. Careful operation of the instrument, selection of proper sample preparation techniques, and good laboratory cleanliness practices are the most important prerequisites for minimizing contamination.

1.5 Definition of Terms

1.5.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

1.5.2 **LFB/BS** - Laboratory Fortified Blank/Blank Spike - A sample of DIW to which a known amount of Hg is added.

1.5.3 **CRA** – a sample of DIW to which a known amount of Hg is added. This is at a low concentration to verify the curve is valid near the reporting limit of 0.2 µg/L.

1.5.4 **CCB** - Continuing Calibration Blank - This is the same acronym used in the CLP program. This is a blank which is analyzed immediately after the CCV (almost always after every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has maintained a stable baseline.

1.5.5 **CCV** - Continuing calibration verification. - This is the same acronym used in the CLP program. This is a standard analyzed at some prescribed frequency (almost always after

every 10 samples and at the end of the analytical run) during the analysis sequence to determine whether the instrument or system has remained in calibration.

1.5.6 **CLP** - Contract Laboratory Program - The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

1.5.7 **Corr Coef, CC** - Correlation coefficient - A measure of the "goodness of fit" of a set of data to a linear regression model. The closer the value is to 1, the higher the degree of confidence in the correlation

1.5.8 **DIW** - Deionized water - Lab reagent water. This water should be free of virtually all analytes.

1.5.9 **ICB** - Initial calibration blank - This term is borrowed from CLP. An instrument blank is made up in the same way as calibration standards, without target analytes.

1.5.10 **ICV** - Initial calibration verification - is a standard that is analyzed at the start of each analytical run that is compared to the initial multi-point calibration to determine whether the instrument calibration is accurate. This verification standard is from a source different from that used to make the calibration standards

1.5.11 **IDL** - Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of least 3x. Determined quarterly using blanks to which a known quantity of standard has been added. The IDL is determined by analyzing 7 replicates spiked at 2-5 times the expected IDL on three non-consecutive days. The sum of the standard deviations of the three runs is the IDL.

1.5.12 **MDL** - Method detection limit - The lowest concentration a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks annually.

1.5.13 **MDL** standard - Method detection limit standard - A standard prepared so that the concentration of Hg is 1-5 times the anticipated MDL

1.5.14 **LCS** - Laboratory Control Sample - A sample containing a known quantity of Hg used to verify digestion and analysis. LCSW indicates a water sample. It is used interchangeably with ICV. LCSS indicates a soil sample.

1.5.15 **RSD or %RSD** - Relative standard deviation or percent relative standard deviation - The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.

1.5.16 **PBW** - A blank carried through the sample preparation and digest procedures.

2 Equipment, Standards and Reagents

2.1 Equipment

2.1.1 Equivalent equipment may be used where appropriate.

IBM Computer and monitor
AA Winlab 32 Instrument Control, Software Version 6.2.0.0079
Perkin Elmer FIMS 400
Perkin Elmer FIMS 100
Perkin Elmer AS-90 or AS-91 autosampler
HP LaserJet4 laser printer
CPI International MOD block with 33 holes (for 120 mL vials)
120 mL Plastic vials with snap seal
Analytical balance
Micropipettes: 20, 50, 100, 200, 500 and 1000 µl
50 mL and 15 mL plastic centrifuge tubes

2.2 Standards

2.2.1 All standards must be prepared in 2.5% HNO_3.

2.2.2 **1000 ppm Hg stock solution**: Supplied currently by CPI International.

2.2.3 **Working Hg solution**: Dilute 100 µl of 1000 ppm Hg stock solution to 200 mL in DIW to which 5.0 mL HNO_3 has been added. The concentration of this solution will be 0.5 ppm Hg. The identification number of the standard solution used and the dilution must be entered in the standards logbook. Expiration date of solution is 6 months from the date of preparation, or the date at which the parent stock solution expires, whichever is sooner. The identification number of the working standard must also be recorded on the bench sheet (digestion log-Appendix III).

2.2.4 **0.2 µg/L standard** – For waters, place 50 mL DIW in a plastic vial. Add 20 µl working Hg solution. For soils, place 10 mL DIW in a plastic vial. Add 40 µl working Hg solution. (Note: Final volume of soils standards after digestion will be 100 mL, waters 50 mL)

2.2.5 **0.5 µg/L standard** - For waters, place 50 mL DIW in a plastic vial. Add 50 µl working Hg solution. For soils, place 10 mL DIW in a plastic vial. Add 100 µl working Hg solution.

2.2.6 **1.0 µg/L standard** - For waters, place 50 mL DIW in a plastic vial. Add 100 µl working Hg solution. For soils, place 10 mL DIW in a plastic vial. Add 200 µl working Hg solution.

2.2.7 **2.0 µg/L standard** - For waters, place 50 mL DIW in a plastic vial. Add 200 µl working Hg solution. For soils, place 10 mL DIW in a plastic vial. Add 400 µl working Hg solution.

2.2.8 **5.0 µg/L standard** - For waters, place 50 mL DIW in a plastic vial. Add 500 µl working Hg solution. For soils, place 10 mL DIW in a plastic vial. Add 1000 µl working Hg solution.

2.2.9 **10.0 µg/L standard** - For waters, place 50 mL DIW in a plastic vial. Add 1000 µl working Hg solution. For soils, place 10 mL DIW in a plastic vial. Add 2000 µl working Hg solution.

2.2.10 **100 ppm Hg stock solution**: Vendor supplied. Currently purchased from Inorganic Ventures, Inc.

2.2.11 **ICV Standard** - Dilute 100µl of 1000 ppm Hg secondary stock solution to 200 mL in DIW to which 5.0 mL HNO_3 has been added. The concentration of this solution will be 0.5 ppm Hg. The identification number of the standard solution used and the dilution must be entered in the standards logbook and the digestion log (Appendix III). Expiration date of solution is 6 months from the date of preparation, or the date at which the parent stock solution expires, whichever is sooner.

2.2.12 **ICV solution** – For waters, a place 50 mls DIW into a plastic vial. For soils, place 10 mL DIW in a plastic vial. Add sufficient ICV solution to produce a concentration of 1.0 to 5.0 µg/L. Currently, we add 400 µL (water sample)/800 µL (soil sample) to produce a concentration of 4.0 µg/L.

2.2.13 **CCV/CCB/Blank Spike (Soils only)** - In setting up the run, use the 5.0 µg/L standard for the CCV and Blank Spike; for the CCB use the calibration blank.

2.2.14 **MS/MSD or MS/Duplicate** - Add 500 µl (water sample)/1000 µL (soil sample) of the working Hg solution to the samples being spiked. When performing Hg under CLP

protocols use 100 µl (water sample)/200 µL (soil sample) of the working Hg solution and perform MS/Duplicate.

2.2.15 **LCSS** - Laboratory control soil sample. Concentrations and limits are certified by the vendor.

2.3 Reagents:

2.3.1 DIW - deionized water free of impurities.

2.3.2 H_2SO_4 (sulfuric acid) - concentrated, trace metal grade

2.3.3 HNO_3 (nitric acid) - concentrated, trace metal grade

2.3.4 HCl (hydrochloric acid) - concentrated, trace metal grade

2.3.5 Stannous chloride - Add 27.5 g $SnCl_2 \cdot 2H_2O$ (stannous chloride dihydrate) to 2.5 L DIW to which has been added 75 mL concentrated HCl. Solution is stored in a glass bottle and is good for 6 months.

2.3.6 $KMnO_4$ (potassium permanganate) - Dissolve 100 g $KMnO_4$, marked "suitable for Hg determination", in 2 L DIW. This is a saturated solution. Sufficient undissolved $KMnO_4$ should be allowed to remain in the bottom of the bottle to assure a saturated solution. Solution is stored in an amber glass bottle and is good for six months.

2.3.7 $K_2S_2O_8$ (potassium persulfate) - Dissolve 50 g $K_2S_2O_8$, marked "suitable for Hg determination", in 1 L DIW. This is a saturated solution. Sufficient undissolved $K_2S_2O_8$ should remain in the bottom of the bottle to assure a saturated solution. Solution is stored in an amber glass bottle and is good for six months.

2.3.8 $NaCl/NH_2OH \cdot HCl$ (sodium chloride/hydroxylamine hydrochloride) - Dissolve 240 g NaCl (table salt is generally used) and 240 g $NaCl/NH_2OH \cdot HCl$ (reagent grade marked "suitable for Hg determination") in 2 L DIW. Contamination has been experienced with this solution. Solution is stored in a glass bottle and is good for 6 months.

2.3.9 Carrier solution - The carrier is a 3% HCl solution. Add 75 mL conc. HCl to 2 L of DIW. Dilute to a final volume of 2.5 L. Solution is stored in a glass bottle and is good for 6 months.

3 Safety Precautions and Waste Disposal

3.1 Safety Precautions

3.1.1 All standards, samples and sample solutions should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Because of the toxic nature of Hg vapor, inhalation or skin contact should be avoided.

3.1.6 The usual precautions should be taken in handling acids.

3.1.7 $SnCl_2$ is a skin and eye irritant; avoid contact.

3.1.8 The addition of hydroxylamine after digestion may release gas including chlorine. Avoid inhalation; use hood if necessary.

3.1.9 Waste disposal – See SOP LTL-2001 for general information on waste disposal. In addition, sample digestates are acidic in nature and must be neutralized prior to disposal.

4 Calibration and Quality Control

4.1 Method Detection Limit Study (MDL).

4.1.1 The estimated MDL for waters is currently 0.10 µg/L. The estimated MDL for soils is currently 0.04 mg/kg. Current MDL values are found in the iLIMS database, with associated raw data stored in department files.

4.1.2 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in Laucks SOP on MDL studies. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL. MDL studies for Hg water samples and soil samples are performed annually.

4.2 Initial Multi-Point Calibration:

4.2.1 Concentrations:

Standard Name	Concentration
S0	0.0 µg/L
S0.2	0.2 µg/L
S0.5	0.5 µg/L
S1.0	1.0 µg/L
S2.0	2.0 µg/L
S5.0	5.0 µg/L
S10.	10.0 µg/L

Standards are prepared fresh daily from the working standard in 2.5% HNO_3. See section 2.2.2 through 2.2.9 for the preparation details.

4.2.2 The calibration blank, the ICV, the LCS, the LFB/BS, the ICB and all standards are digested along with the samples being analyzed.

4.3 Calibration Criteria

4.3.1 Calibration curve – "best fit" of points in calibration curve. Linear correlation coefficient r must be equal to or greater than 0.995.

4.3.2 ICV - This is run immediately following a successful curve. The ICV is made from a source of mercury independent from the calibration source and is used to verify the curve is valid. For EPA 245.1/245.5 analyses the recovery limits are 95-105 %, for SW-846 7470/7471 the limits are 90-110%, and for CLP the limits are 80-120%. If recovery is outside of the control limits an immediate reanalysis is permitted. If still out of control the curve must be reanalyzed followed by another ICV otherwise continue with an ICB.

4.3.3 ICB – This is run immediately following a successful ICV. The ICB is used to verify that there is no contamination and that the curve is valid. Control limits presently in use for EPA 245.1/245.5 and SW-846 7470/7471 are ±0.1 µg/L (1/2 of the reporting limit), for CLP they are ±0.2 µg/L (reporting limit). If recovery is outside of the control limits an immediate reanalysis is permitted. If still out of control, a corrective action must follow including a reanalysis of the curve.

4.3.4 A PBW and an LFB/BS are run immediately following the successful calibration. The control limits for the PBW are the same as those used for the ICB. The control limits for the LFB are as set forth in the individual methods and in the laboratory QC database.

4.3.5 If the above criteria are not met, recalibration must be performed.

4.4 Continuing Calibration:

4.4.1 A CCV followed immediately by a CCB must be run every 10 samples and after the last sample. The ICB, PBW and LFB/BS count as samples for this purpose. Periodically, the CCV must be analyzed at a different level within the calibration range than routinely analyzed. A copy of this CCV must be available for review.

4.4.2 Criteria - For CCV's, the recovery limits are 80% to 120% (90-110% for 245.1/245.5). For CCBs, the control limits are ±0.1 µg/L (±0.2 µg/L for CLP).

4.4.3 Corrective action - If a CCV or CCB is out of control, an immediate reanalysis is permitted. However, if the reanalysis is out of control a reanalysis of all samples since the previous successful CCV and/or CCB must be performed.

4.5 Matrix Spike

4.5.1 A sample is chosen at random from the samples to be analyzed, and 500 µl of the working standard is added (100µl for CLP). The spike level is then 5.0 µg/L (1.0 µg/L for CLP). The analyst should attempt to avoid selecting samples that are identified by the client as blanks. As the purpose of the matrix spike is to test the system under "typical" conditions, the analyst may also avoid selecting the most difficult sample of the batch for spiking. It is not always required that a matrix spike analysis be performed with each preparation/analysis batch, however, the minimum frequency for MS analysis is 1 each per 20 samples per matrix. This will be best accomplished by running one with every batch for many analyses. This matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$\text{recovery, \%} = \frac{(SS - S)}{SA} * 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample
SA = spiked added, the amount of spiking material actually added to the sample calculated on the sample basis

4.5.2 The recovery criteria are listed in the quality control database and will change from time to time. Current acceptable recovery limits are 85-115% for SW-7470/7471, 70-130% for EPA 245.1/245.5 and 75-125% for CLP.

4.5.3 Corrective action

4.5.3.1 Samples with spike recoveries outside control limits will be reviewed for possible corrective action. Corrective action will first involve recalculation, followed by possible re-preparation, and/or reanalysis. This process should also look at the recovery of Hg from the LCS and/or blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

4.6 Matrix Spike Duplicate

4.6.1 The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} * 100$$

where:
S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

4.6.2 RPD control limits are listed in the quality control database and will change from time to time. The currently acceptable RPD limit is 20%.

4.7 Sample Duplicate

4.7.1 Sample duplicates are required when CLP methods are employed, or when the method specifically calls for duplicates. At least one duplicate sample per 20 samples per matrix is required when matrix spikes are being performed.

$$RPD = \frac{|S1 - S2|}{(S1 + S2)/2} * 100$$

where:
S1 = measured concentration in the initial analysis
S2 = measured concentration in the duplicate analyses

4.7.2 The RPD control limits listed in the quality control database and will change from time to time. The current acceptable RPD limit is 20%.

4.7.3 Corrective action

4.7.3.1 If a trend in out of control RPD values is observed, the methods used must be examined to determine the source of variance. Once this source is identified, corrective action, up to and possibly including changes to the method, must be performed so that samples can be analyzed with a predictable reproducibility. Generally, if recoveries are in control and no analyte of interest was detected in any of the samples, no immediate action will be taken on that sample set. If integrity of reported sample values is in doubt, re-analysis may be called for. Corrective actions should be discussed with the Quality Assurance Officer.

5 Operation Procedures

5.1 MOD Block – Plastic vial – Water or Soil (standard preparation):

5.1.1 Digestion log (Appendix III): The digestion log should list the sample numbers and bottle numbers, sample volume or weight, the date, the name of the analyst, the time placed in the MOD block, the time when the temperature reached 95 ± 5° C, and the time removed from the MOD block. One should be prepared for each run. An example is attached as Appendix III. The numbers of each plastic vial placed in the leftmost column of this form to identify which sample is in each plastic vial.

5.1.2 Turn on MOD block. The MOD block can be operated in either Manual or Automatic mode. The manual mode provides continuous temperature control while the Automatic mode provides a timed temperature program. Manual mode is utilized for mercury digestions to provide a consistent internal sample temperature.

5.1.2.1 Set Manual mode by pressing Auto/Manual until the light above the manual mode indicator is lit.

5.1.2.2 Press ENTER to begin programming. Press the UP arrow to set temperature to 132°C. This will result in an internal temperature of approximately 95°C. Press Enter once again to begin heating. The block Status indicator will flash until the block reaches the set temperature.

5.1.3 Laucks Testing Laboratories has studied the accuracy in using the MOD Block Digestor in place of the water bath for sample digestion. Due to digestion vessel volume limitations, 50 mL of sample is used. MDL studies have found results comparable to the water bath in all methods.

5.1.4 Preparation of standards: See section 2.2.

5.1.4.1 The standards, the ICV and the ICB must be digested along with the PBW, LFB/Blank Spike and samples.

5.2 MOD Block Digestion – Water Samples

5.2.1 These procedures are for methods SW-846 7470, CLP ILM0 4.1, ILM05.3 and EPA 245.1 unless noted otherwise.

5.2.2 Place 50 mL sample in the plastic vial designated for that sample, being sure to place 50 mL sample in the designated duplicate, MS and MSD plastic vials for the sample involved. TCLP samples should be diluted 10 mL/50 mL (dilution factor of 5) in DIW. TCLP spikes should be diluted 1 mL/50 mL (dilution factor of 50) in DIW. Record all dilutions on the digestion log.

5.2.3 Add the required quantity (see Sec. 2.2.14) of the working standard to the MS and MSD plastic vial.

5.2.4 Add the following reagents to each plastic vial (for all methods):

2.5 mL concentrated H_2SO_4
1.25 mL concentrated HNO_3
8 mL $KMnO_4$ solution
4 mL $K_2S_2O_8$ solution

5.2.5 The $KMnO_4$ color must persist for 15 minutes. If it does not, additional 4 or 8 mL aliquots of KMnO4 must be added until color persists.

5.2.5.1 Snap the plastic vial lids down loosely to allow steam to escape and prevent the buildup of pressure. Lids should cover the samples and allow fluid to reflux without building pressure. Alternatively a ribbed watchglass can be used to cover the sample vials.

5.2.5.2 Place all plastic vials, being careful to keep them in order, in the MOD block in a hood. The Status indicator light will begin flashing as the block reheats to its set temperature after addition of the vials. Once the block set temperature returns to the set 132°C (the Status indicator will stop flashing), record the time and temperature of samples and continue heating for two (2) hours.

5.2.5.3 Remove the plastic vials from the block and allow to cool.

5.2.5.4 Add 4 mL $NaCl/NH_2OH{\cdot}HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear on standing after a few

minutes. Bring the final volume of each standard and digest to 50 mL by the addition of DIW using the graduated marks on each vial.

5.3 MOD Block Digestion – Soil Samples

5.3.1.1 These procedures are for Methods SW-846 7471 and EPA 245.5 unless noted otherwise:

5.3.1.2 Weigh approximately 0.5 g (or for certain clients, approximately 0.2 g or 2.5 g) of sample into each properly marked 120 mL vial. Record the exact weight on the digestion log bench sheet.

5.3.1.3 Add 10 mL of DIW to each sample vial containing soil. Add 3.75 mL concentrated HCl and 1.25 mL concentrated HNO_3 to each sample and standard vial (for ILM04.1, add 5.0 mL concentrated H_2SO_4 and 2.5 mL (2.0 mL for ILM05.3) of concentrated HNO_3).

5.3.1.4 Place acidified soil sample vials in preheated MOD block. MOD block controller should read a set temperature of 132°C. Maintain the heat at this level for 2 minutes.

5.3.1.5 Cool and add 40 mL DIW and 15 mL $KMnO_4$ to all sample and standard vials (also add 8 mL $K_2S_2O_8$ for CLP). Mix.

5.3.1.6 The $KMnO_4$ color must persist for 15 minutes. If it does not, more $KMnO_4$ must be added.

5.3.1.7 Snap the plastic vial lids down loosely to allow steam to escape and prevent the buildup of pressure. Lids should cover the samples, yet allow fluid to reflux without building up pressure. Alternatively a ribbed watchglass can be used to cover the samples and standards.

5.3.1.8 Place all plastic vials, being careful to keep them in order, in the MOD block in a hood. The Status indicator light will begin flashing as the block reheats to its set temperature after addition of the vials. Once the block set temperature returns to the set 132°C (the Status indicator will stop flashing), record the time and continue heating for 30 minutes.

5.3.1.9 Remove the plastic vials from the block and allow to cool.

5.3.1.10 Add 7 mL $HCl/NH_4NH_2OH.HCl$ solution and mix until the mixture clears. If some undissolved permanganate remains, it will usually disappear upon standing for a few minutes. Bring the final volume of each standard and digest to 100 mL by the addition of DIW using the graduated marks on the vial.

5.4 Analysis on FIMS 400 or FIMS 100

5.4.1 Both instruments are operated using the same software program, WinLab 32 Instrument Control, Version 6.2.0.0079, copyright 2003. This is a programmed analysis using a 6-point curve with a blank correction. The development of the calibration curve, including the ICV and ICB, is automatic. If the calibration is unsuccessful, recalibration is attempted. The 5.0 µg/L standard is used for CCV's; the calibration blank for CCB's; both of which are programmed to be run every 10 samples. If a CCV or a CCB fails to meet quality control specifications, the system stops and all samples since the last preceding valid CCV or CCB are reanalyzed. A CCV and a CCB are also run after the last sample in the run.

5.4.2 Open the 'WinLab 32 for AA' icon on the desktop. Then click the 'Workspace' icon and open default.ffm. This should present four windows on the screen for Sample information, FIAS control, Automated Analysis control, and Results.

5.4.3 Go to File, open, sample info file and open forms.sif for soils or formw.sif for water samples. Fill in the batch ID field using the date the samples were digested, the sample matrix and the instrument used (i.e., for 090105w.400, the samples would have been digested on 9/01/05, be water samples, and be analyzed on the FIMS 400). Fill in the sample ID's, initial weights or volumes and sample prep volumes. Do not include calibrators in the sequence. These will be inserted at the appropriate intervals by the selected method (see below). Go to File, save as, sample info file and save using the same name as was used for the batch ID. Print a copy of the sample info file by going to File, print active window.

5.4.4 All autosampler tubes should be marked with their contents before being filled. Then the tubes are filled from the plastic digestion vials and placed in the autosampler locations designated either by the method or by the sample information sheet.

5.4.5 Click the 'Method Editor' icon. Open the water or soil method as needed. On the opened method, click the 'OK QC' tab and then the 'Schedule QC' tab. Ensure that the calibrators are scheduled correctly.

5.4.6 Go to the 'FIAS Control' window. For the FIMS 400, use the pump 2 icon to check the flow rates for the carrier solution and the stannous chloride solution. For the FIMS 100, use the pump 1 icon to check the flow rates. The flow rate for the carrier solution should be between 4-6 mL per 30 seconds. The flow rate for the stannous chloride solution should be between 2-3 mL per 30 seconds. Record results in the logbook.

5.4.7 Go to the 'Automated Analysis Control' window. Click the 'setup' tab. Unclick the box for 'lamps off at the end of the analysis'. Click on the box for 'print log during analysis'. Click on the box for 'pumps off at end of analysis'.

5.4.7.1 Click the 'analysis' tab. Place 200 µL of the ICV 0.5 ppm standard in an autosampler tube and dilute to 10 mL. Mix well by inversion. Place tube in the last position in the sampler rack. Click 'select loc' button, type in the location of the tube and click 'OK'. Click 'Results' window and check the absorbance. The absorbance should be between 0.12 and 0.25. Record results in the logbook.

5.4.7.2 Go to the 'Automated Analysis Control' window. On the 'setup' tab, check that the sample info file listed in the field is correct. For the results data set field, click open and enter the same name as the sample info file. Enter the description of the date digested, the matrix and the SDG names (if desired). Click OK. Back on the 'setup' tab, ensure that the save data and sample info boxes are checked on.

5.4.7.3 If this is to be the last analytical run of the day, ensure that the lamp will be turned off at the end of the analysis. This can be done at the 'setup' tab or it can be done manually from the analysis menu at the top of the screen.

5.4.7.4 Go to the 'analyze' tab. Click 'rebuild list' button. Check that the sequence is correct. Click 'analyze all' button. The 'priority' button can be used to attach additional samples to the analytical run. This can be used to reanalyze questionable results or to analyze diluted samples.

5.4.7.4.1 Dilution - Where analysis results are above the high standard (10.0 µg/L) an aliquot of the sample diluted with the calibration blank will be analyzed.

5.5 Reporting results from the FIMS 400 or FIMS 100.

5.5.1 Open the 'Data Manager' icon on the desktop. Go to File, select library, and open the results folder for the month desired. Highlight the file that you wish to report

5.5.2 Click the 'Report' icon. Click 'use existing design'. Use the 'browse' button to select the report format desired and open it. Click the 'next' button to page forward. On page 2, you can enable / disable the injections to be reported. On page 5, enter the method, instrument, date analyzed, and analyzed by in the appropriate fields. On page 8, click 'preview' and check the report on the screen. Then print the report. Do not save the design. Click cancel to close the window.

5.6 Exporting results from the FIMS 400 or FIMS 100.

5.6.1 Go to File, select library, and open the results folder for the month desired. Highlight the file that you wish to export.

5.6.2 Click the 'Export' icon. Click 'use existing design'. Use the 'browse' button to select the report format desired and open it. For the FIMS 400, use file marrs.xpt. For the FIMS 100, use the file marrs1.xpt. Click the 'next' button to page forward. On page 2, you can enable / disable the injections to be reported. On page 4, check that the file will be sent to p:\metals\mercury\Data from FIMS \Reports. On page 8, click 'export data'. Do not save the design. Click cancel to close the window.

5.6.2.1 Files from the instrument are backed up onto the network on a monthly basis.

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 The final data packet is to include the following:

1) The report (See Appendix IV).
2) The digestion log (Appendix III).
3) The Sample Information Detail Report
4) The raw data
5) The quality control database form

7.1.2 All results for quality control tests are entered into the quality control database. A summary report of all data entered must be included in the data packet. The routine minimum is a method blank report, a method blank spike, an MS/MSD or MS/duplicate report and a SRM/LCS.

7.2 Data Qualifying Flags

CODE	Definition	
U	The analyte of interest was not detected, to the limit of detection indicated.	❁◆
B	The analyte of interest was detected between the IDL/MDL and the reporting limit.	❁
J	The analyte of interest was detected between the MDL and the reporting limit. (ILM05.3)	❁
N	The spike recovery exceeded the control limits.	❁
*	The duplicates exceeded the RPD control limit or their difference exceeded the reporting limit.	❁

◆ Used in all reports.
❁ Used in data validatable packages.
Other flags may be used and will be specified on a project-specific basis.

8 References

Test Methods for Evaluating Solid Waste Physical/Chemical Methods, USEPA SW846, Method 7470A/7471A, September 1994

Determination of Mercury in Water by cold vapor atomic absorption spectrometry, USEPA 245.1/245.5, most recent version, Method 245.1/245.5

Department Of Defense Quality Systems Manual for Environmental Laboratories, Version 3, March 2005

Air Force Center for Environmental Excellence (AFCEE) Quality Assurance Project Plan, Version 4.0.01, May 2005

USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, ILM04.1, Jan. 2000

USEPA Contract Laboratory Program Statement of Work for Inorganic Analysis, ILM05.3, Dec. 2001

FIMS Flow Injection Mercury System, Perkin Elmer, Installation Maintenance System Description, Release 1.1, June 1994

FIMS Flow Injection Mercury System, Perkin Elmer, Setting Up and Performing Analyses, Release 1.1, March 1994, Release C, July 2003

FIMS Flow Injection Mercury System, Perkin Elmer, Hardware Guide, Release C, January 2002

Flow Injection Mercury / Hydride Analysis, Perkin Elmer, Recommended Analytical Conditions and General Information, Release 3.0, February 1994, Release 5.0, July 2000

AS-90 Autosampler for Atomic Spectroscopy, Perkin Elmer, Installation, Maintenance and System Description, Release 4.0, April 1994

Appendix I - QC Summary Table

Laucks Testing Laboratories
Method 245.1/245.5, 7470/7471, & CLP ILM04.1/05.3 QA Requirements and Corrective Actions

(See Appendix II for additional Agency Specific Criteria)

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Initial Calibration	r > 0.995	r > 0.995 6-point curve + blank	Once per day, per matrix	Recalibrate	Run log
Initial Calibration Verification	SW846 and DOD-QSM 90-110%, EPA 245 95-105%, and CLP 80-120%	SW846 and DOD-QSM 90-110%, EPA 245 95-105%, and CLP 80-120%	Once per batch	Recalibrate	Run log, report
Initial Calibration Blank	Below MDL	Below ½ R.L.	One per batch	Recalibrate	Run log, report
Method Blank (PBW)	Below R.L.	Below ½ R.L.	One per batch	Consult QC officer. Flag data or reanalyze.	Quality control database
Continuing Calibration Verification	SW-846 7470/7471 80-120%, and EPA 245 90-110%	SW-846 7470/7471 80-120%, and EPA 245 90-110%	Every 10 samples and after the last sample	Rerun samples since last CCV	Run log
Continuing Calibration Blank	Below MDL	Below ½ R.L.	Every 10 samples and after the last sample	Rerun samples since last CCB	Run log
Matrix Spike Recovery	SW-846 7470/7471/DOD-QSM 80-120%, EPA 245.1/245.5 (70-130%). CLP Low/CLP (75-125%)	SW-846 7470/7471/DOD-QSM 80-120%, EPA 245.1/245.5 (70-130%), CLP Low/CLP (75-125%)	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
MS/MSD RPD	20% RPD	20% RPD	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database
Duplicate % Difference	20% RPD	20% RPD	Every 20 samples	Consult QC officer. Flag data or reanalyze.	Quality control database
Standard Reference Material (LCS) Recovery	Soil: Vendor's limits Water: See ICV	Soil: Vendor's limits Water: See ICV	Once per 20	Recalibrate	Run log

Appendix II - Agency Criteria

Study/QC	SW-846 7470/7471		DOD-QSM (Ver. 3)		AFCEE (4.0)		ILM 4.1		ILM 5.3		245.1/245.5	
	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency
IDLs	No criteria	---	No criteria	---	---	---	Initially	3 mo.	---	---	N\A	N\A
MDLs	No criteria	---	N\A	Initially & once per 12 mo. Quarterly MDL checks may be performed instead.	<=1/2 RL	Initially & once per 12 mo. or quarterly MDL verification checks	N/A	N/A	<1/2 CRQL	Initially & every 12 mo.	No criteria	12 mo.
Linear Range Study	No criteria	---	N\A	---	---	---	N/A	N/A	---	Initially	+/- 10%	Initially
Linear Range Check Std.	No criteria	---	N\A	---	---	---	N/A	N/A	+/- 5%	3 mo.	+/- 10%	12 mo
ICAL	Minimum of 3 standards and a blank.	---	Minimum of 5 standards and a blank. r>=0.995	---	Minimum of 5 standards and a blank. r>=0.995	Daily prior to sample analysis	Minimum of 3 standards and a blank. One standard at the CRDL. r>=0.995	Daily	Minimum of 5 standards (one at the CRQL) and a blank. Each standard shall be within 5% of true value except std at CRQL. r>=0.995		No criteria	Daily
ICV/CCV	+/- 10% (ICV). '+/- 20% (CCV).	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)	+/- 10% (ICV). '+/- 20% (CCV).	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)	+/- 10% (ICV). '+/- 20% (CCV).	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)	+\- 20%	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)	+/- 20%	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)	ICV +/- 5%, CCV +/- 10% (after initial CCV)	Immed. after calib.(ICV), after every 10 samples (CCV), and end of run (CCV)
ICB/CCB/PB	No criteria	---	No analytes detected > 2x MDL (ICB/CCB). No analytes detected >1/2 RL (PB)	Immed. after calib.(ICB), after every 10 samples (CCB), and 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), after every 10 samples (CCB), and end of run (CCB). One per analytical batch (PB)	<=CRDL	Immed. after calib.(ICB), after every 10 samples (CCB), and end of run (CCB). One per analytical batch (PB)	<= CRQL	Immed. after calib.(ICB), after every 10 samples (CCB), and end of run (CCB). One per analytical batch (PB)	< MDL (ICB/CCB). <2.2xMDL or <10% of sample level(PB)	Immed. after calib., after every 10 samples, and end of run (ICB/CCB). One in 20 per batch per matrix (PB)
Low Level Check Std. (CRI/CRDL/CRQL/CRA)	No criteria	---	N\A	---	No criteria	---	No criteria	Immed. after calib (ICB). CRA at the CRDL	+/- 30%.	Beginning of run (after ICV), every 20 samples and end of run. CRI to be immediately followed by CCV/CCB. "CRI" at the CRQL	N\A	---

Study/Q	SW-846 7470/7471		DOD-QSM (Ver. 3)		AFCEE (4		ILM 4.1		ILM 5.3		24 .5.5	
	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency	Criteria	Frequency
LCS (BS)	No criteria	One in 20 per prep batch	80-120% (per DoD-QSM Appendix DoD-D)	One per prep batch	80-120%	One per prep batch	H_20: N/A. Soil: Per vendor limits.	H_20: Not required. Soil: One per prep batch	H_20: N/A. Soil: Per vendor limits.	One per prep batch per matrix	85-115%	One per prep batch per matrix
Matrix Spike (MS)	No criteria	One in 20 per prep batch per matrix	Per DoD LCS limits	One per prep batch per matrix	H_2O & Soil: 80-120%.	One MS/MSD per 20 samples	75-125%	One per prep batch	75-125%, unless sample level >4x spike level	One per prep batch per matrix (1 ug/L for H_20, 0.5 mg/kg for soil)	70-130%	10% of samples (MS/MSD per 20 samples)
Duplicate (D) or Matrix Spike Duplicate (MSD)	No criteria	One in 20 per prep batch per matrix (MSD)	RPD <= 20%	One per prep batch per matrix	Matrix Spike:: H_2O: RPD <= 15%, Soil: <=30%	One MS/MSD per 20 samples (No Duplicate)	RPD <= 20% if analyte level >=5x CRDL. +/- CRDL, otherwise.	One per prep batch (D)	RPD <= 20% if analyte level >=5x CRQL. +/-CRQL, otherwise.	One per prep batch per matrix (D)	N\A	N\A
Serial Dilution (L)	% Diff <= 10% (Only for sample concs. >25x MDL)	One per prep batch	% Diff <= 10% (Only for sample concs. >25x MDL)	One per prep batch	% Diff <= 10% (Only for sample concs. >=25x MDL)	One per prep batch per matrix	N/A	N/A	N\A	N\A	N\A	N\A
Post Digestion Spike (PS)	No criteria	---	No criteria	---	No criteria	---	N/A	N/A	N\A	N\A	N\A	N\A

Appendix III – Sample Digestion Log

Mercury Digestion Log - Waters

SW-846 7470, EPA 245.1

Note: All samples are digested according to SW-846 7470 unless otherwise stated in the sample comments

Date		Vials into of MOD BLOCK (time)	
Analyst		Vials out of MOD BLOCK (time)	
Standard ID No.	ME-15-	ICV Standard No.	ME-15-
	Sulfuric	Nitric	Hydrochloric
Acid Lot #			
Temperature at start of digestion (°C)		Temperature at end of digestion(°C)	

No.	Sample ID	Sample size (mL)	Final volume (mL)	Comments	Bottle #
1	S0 ppb		50		
2	S0.2 ppb 20µl of Std.		50		
3	S0.5 ppb 50µl of Std.		50		
4	S1.0 ppb 100µl of Std.		50		
5	S2.0 ppb 200µl of Std.		50		
6	S5.0 ppb 500µl of Std.		50		
7	S10.0 ppb 1000µl of Std.		50		
8	ICV 400µl of ICV		50		
9	ICB		50		
10	LCSW		50	400µl of ICV	
	CRA 20µl of Std.LCSW		50		
11	B		50		
12			50		
13			50		
14			50		
15			50		
16			50		
17			50		
18			50		
19			50		
20			50		
21			50		
22			50		
23			50		
24			50		
25			50		
26			50		
27			50		
28			50		
29			50		
30			50		
31			50		
32			50		
33			50		

Mercury Digestion Log - Waters

ILM04.1

Note: All samples are digested according to ILM04.1 unless otherwise stated in the sample comments

Date		Vials into MOD BLOCK (time)	
Analyst		Vials out of MOD BLOCK (time)	
Standard ID No.	ME-15-	ICV Standard No.	ME-15-
	Sulfuric	Nitric	Hydrochloric
Acid Lot #			
Temperature at start of digestion (°C)		Temperature at end of digestion(°C)	

No.	Sample ID	Sample size (mL)	Final volume (mL)	Comments	Bottle #
1	S0 ppb		50		
2	S0.2 ppb 20µl of Std.		50		
3	S0.5 ppb 50µl of Std.		50		
4	S1.0 ppb 100µl of Std.		50		
5	S2.0 ppb 200µl of Std.		50		
6	S5.0 ppb 500µl of Std.		50		
7	S10.0 ppb 1000µl of Std.		50		
8	ICV 400µl of ICV		50		
9	ICB		50		
10	LCSW		50	100µl of ICV	
	CRA 20µl of Std.		50		
11	B		50		
12			50		
13			50		
14			50		
15			50		
16			50		
17			50		
18			50		
19			50		
20			50		
21			50		
22			50		
23			50		
24			50		
25			50		
26			50		
27			50		
28			50		
29			50		
30			50		
31			50		
32			50		
33			50		

Mercury Digestion Log - Soils

SW-846 7471, EPA 245.5

Note: All samples are digested according to SW-846 7470 unless otherwise stated in the sample comments

Date		Vials into of MOD BLOCK (time)	
Analyst		Vials out of MOD BLOCK (time)	
Standard ID No.	ME-15-	ICV Standard No.	ME-15-
	Sulfuric	Nitric	Hydrochloric
Acid Lot #			
Temperature at start of digestion (°C)		Temperature at end of digestion(°C)	

No.	Sample ID	Sample size (mL)	Final volume (mL)	Comments	Bottle #
1	S0 ppb		100		
2	S0.2 ppb 40µl of Std.		100		
3	S0.5 ppb 100µl of Std.		100		
4	S1.0 ppb 200µl of Std.		100		
5	S2.0 ppb 400µl of Std.		100		
6	S5.0 ppb 1000µl of Std.		100		
7	S10.0 ppb 2000µl of Std.		100		
8	ICV 800µl of ICV		100		
9	ICB		100		
	CRA 40µl of Std.LCSW		100		
10	B		100		
11	S		100	1000µl of Std.	
12			100		
13			100		
14			100		
15			100		
16			100		
17			100		
18			100		
19			100		
20			100		
21			100		
22			100		
23			100		
24			100		
25			100		
26			100		
27			100		
28			100		
29			100		
30			100		
31			100		
32			100		
33			100		

Mercury Digestion Log - Soils

ILM04.1

Note: All samples are digested according to ILM04.1 unless otherwise stated in the sample comments

Date		Vials into MOD BLOCK (time)	
Analyst		Vials out of MOD BLOCK (time)	
Standard ID No.	ME-15-	ICV Standard No.	ME-15-
	Sulfuric	Nitric	Hydrochloric
Acid Lot #			
Temperature at start of digestion (°C)		Temperature at end of digestion(°C)	

No.	Sample ID	Sample size (g)	Final volume (mL)	Comments	Jar #
1	S0 ppb		100		
2	S0.2 ppb 40µl of Std.		100		
3	S0.5 ppb 100µl of Std.		100		
4	S1.0 ppb 200µl of Std.		100		
5	S2.0 ppb 400µl of Std.		100		
6	S5.0 ppb 1000µl of Std.		100		
7	S10.0 ppb 2000µl of Std.		100		
8	ICV 800µl of ICV		100		
9	ICB		100		
	CRA 40µl of Std.		100		
10	B		100		
11	S		100	200µl of ICV	
12			100		
13			100		
14			100		
15			100		
16			100		
17			100		
18			100		
19			100		
20			100		
21			100		
22			100		
23			100		
24			100		
25			100		
26			100		
27			100		
28			100		
29			100		
30			100		
31			100		
32			100		

Appendix IV - Example Report Form

HG by SW-846

PE FIMS 100

Operator: MU

Hg in Water by SW-846 7470/EPA 245.1

Laucks Testing Laboratories, Inc.

MU, 8/18/05

SampleID	Analyte	Mean
Calib Blank		
	Hg 253.7	
S 0.2		
	Hg 253.7	[0.2] ug/L
S 0.5		
	Hg 253.7	[0.5] ug/L
S 1.0		
	Hg 253.7	[1.0] ug/L
S 2.0		
	Hg 253.7	[2.0] ug/L
S 5.0		
	Hg 253.7	[5.0] ug/L
S 10.0		
	Hg 253.7	[10.0] ug/L
ICV		
2005/08/18 17:03:33 All analyte(s) passed QC.		
	Hg 253.7	4.0 ug/L
2005/08/18 17:03:33 QC value within limits for Hg 253.7 Recovery = 101.17%		
ICB		
2005/08/18 17:05:55 All analyte(s) passed QC.		
	Hg 253.7	0.0 ug/L
2005/08/18 17:05:55 QC value within limits for Hg 253.7 Recovery = Not calculated		
CRA		
2005/08/18 17:08:15 All analyte(s) passed QC.		
	Hg 253.7	0.2 ug/L
2005/08/18 17:08:15 QC value within limits for Hg 253.7 Recovery = 114.42%		
LCSW1		
	Hg 253.7	3.876 ug/L
B081805hgw01		
	Hg 253.7	0.0200 ug/L
0508322-01		
	Hg 253.7	0.1152 ug/L
0508264-01		
	Hg 253.7	1.107 ug/L
0508264-01 dup		
	Hg 253.7	1.094 ug/L
0508264-01 ms		
	Hg 253.7	184.5 ug/L
B081705TCLP		
	Hg 253.7	0.1611 ug/L
CCV		
2005/08/18 17:27:22 All analyte(s) passed QC.		
	Hg 253.7	4.7 ug/L
2005/08/18 17:27:22 QC value within limits for Hg 253.7 Recovery = 94.65%		
CCB		
2005/08/18 17:29:43 All analyte(s) passed QC.		
	Hg 253.7	0.0 ug/L
2005/08/18 17:29:43 QC value within limits for Hg 253.7 Recovery = Not calculated		

Sample Information Detail Report
Document Name: 0801805w.100

File Description
Waters

Parameters Common to All Samples

Batch ID	B081805
Volume Units	mL
Weight Units	g

Parameters That Vary By Sample

Sample No	A/S Location	Sample ID	Sample Prep. Vol.
1	10	LCSW1	50
2	11	B081805hgw01	50
3	12	0508322-01	50
4	13	0508264-01	50
5	14	0508264-01 dup	50
6	15	0508264-01 ms	50
7	16	B081705TCLP	50

Sample No	Aliquot Volume	Diluted To Vol.	Initial Sample Vol.
1			50
2			50
3			10
4			1
5			1
6			1
7			10

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8000

Title: **Determination of Retention Time Windows**

Revision history:

Number	Date
0	10/30/94
1	1/30/98
2	06/22/00
3	06/20/02
4	07/28/04

Revised by: Gail Rossidivito — Date: 7/29/04
Gail Rossidivito, Senior Chemist

Approved by: Kathy Kreps — Date: 7/29/04
Kathy Kreps, Laboratory Director

Approved by: Harry Romberg — Date: 7-30-04
Harry Romberg, QA Officer

Controlled Document

No. 20 **Assigned to:** TETRA

Table of Contents

1. INTRODUCTION AND SCOPE **3**
1.1 METHOD DESCRIPTION 3
2. DEFINITION OF TERMS AND ACRONYMS **3**
3. EQUIPMENT LIST AND STANDARDS **4**
3.1 EQUIPMENT 4
3.2 STANDARDS 4
4. SAFETY PRECAUTIONS AND WASTE DISPOSAL **4**
4.1 SAFETY PRECAUTIONS 4
4.2 WASTE DISPOSAL 4
5. CALIBRATION AND QUALITY CONTROL **5**
5.1 CALIBRATION 5
6. OPERATION PROCEDURES **5**
6.1 DATA COLLECTION 5
6.2 CALCULATIONS 5
7. APPLICATION TO ROUTINE ANALYTICAL SEQUENCES **8**
7.1 SETTING THE RTW FOR EACH ANALYTICAL SEQUENCE 8
7.2 ANALYST DISCRETION IN ANALYTE IDENTIFICATION 8
7.3 CORRECTIVE ACTIONS 8
8. REPORTS **9**
8.1 ORIGINAL DATA 9
8.2 WORKING COPIES 9
9. REFERENCES: **9**

Controlled Document
No. _____ Assigned to: _____

1. Introduction and Scope

1.1 Method Description

1.1.1 Retention time windows are crucial to the identification of target compounds. 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.

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.

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.

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.

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 CLP, the RTWs are fixed in the Statement of Work. This methodology has been adopted by the laboratory for the determination of pesticides/PCBs by SW-846.

The default retention time window half-width (0.03 minutes) that is called out 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.

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.

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

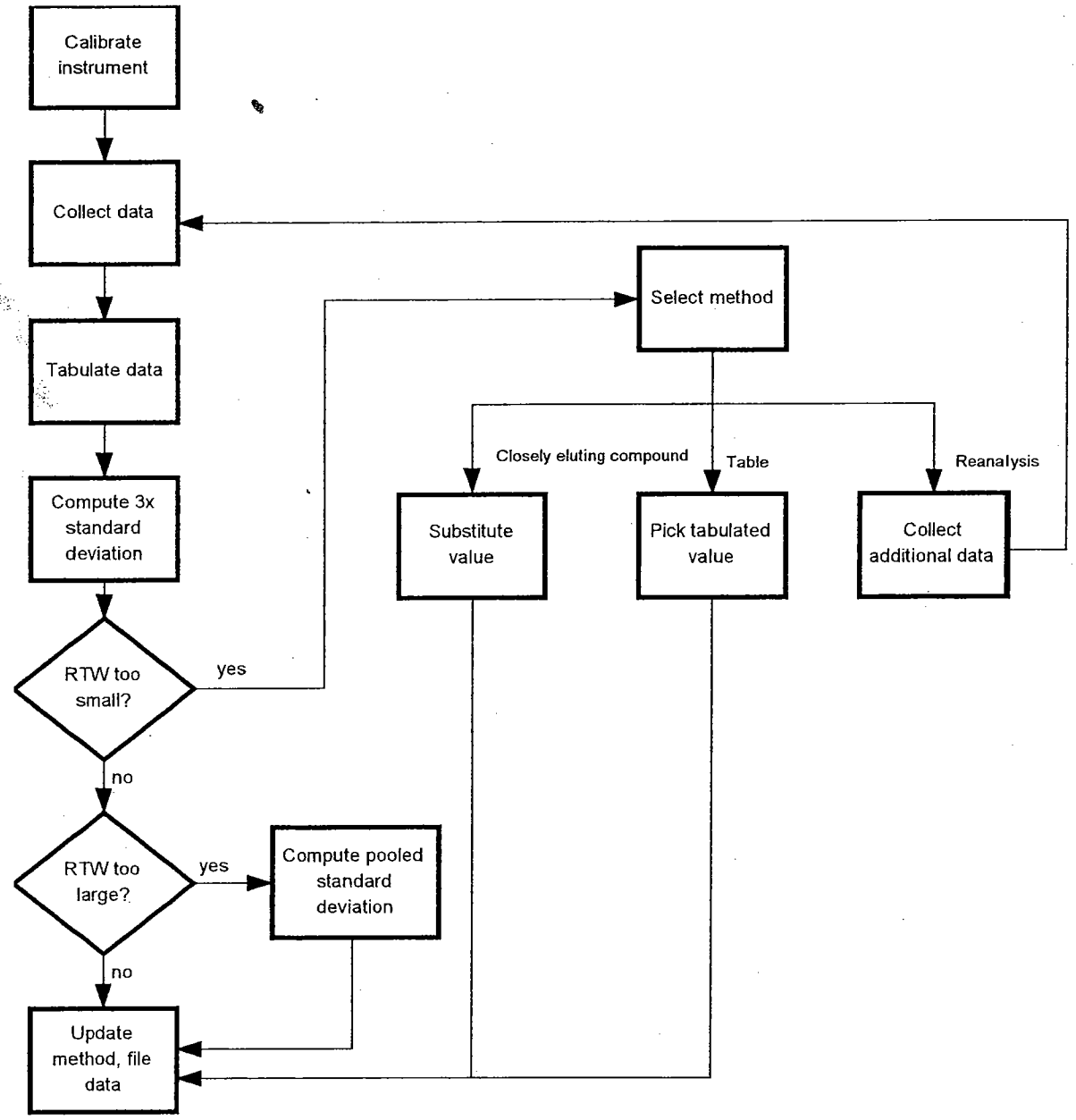
8.2.1 Summarized, tabulated retention time window results will be maintained in the appropriate chromatography laboratory, on an instrument specific basis. On a project specific basis and by request only, these summarized, tabulated RTW results will be provided in the case narrative report.

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

Appendix 1

LTL-8000 Flow Chart



LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8084

Title: **Analysis of Organochlorine Pesticides and PCBs by SW846 Methods 8081B and 8082A**

Revision history:

Number	Date
0	2/11/98
1	7/10/98
2	4/29/99
3	4/27/00
4	9/11/01
5	03/08/02
6	05/30/02
7	09/25/02
8	04/06/05

Revised by: Monica Carr, Organic Division Manager Date: 4/26/05

Approved by: Kathy Kreps, Laboratory Director Date: 4-27-05

Approved by: Harry Romberg, QA Officer Date: 4-26-05

Controlled Document

No. 20 Assigned to: Tetra

TABLE OF CONTENTS

1 INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
SW8082A OPTIONAL COMPOUNDS 4
1.2 METHOD DEVIATIONS 5
1.3 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 5
1.4 DEFINITION OF TERMS 5

2 EQUIPMENT LIST, MAINTENANCE, AND STANDARDS 8

2.1 CHROMATOGRAPHIC AND DATA SYSTEM 8
2.2 STANDARDS 8

3 SAFETY PRECAUTIONS AND WASTE DISPOSAL 10

3.1 ROUTINE SAFETY PRECAUTIONS 10
3.2 WASTE DISPOSAL 10

4 CALIBRATION AND QUALITY CONTROL 10

4.1 QA REQUIREMENTS AND CORRECTIVE ACTION 10
4.2 METHOD DETECTION LIMIT STUDY 10
4.3 METHOD VALIDATION 10
4.4 RETENTION TIME WINDOWS 11
4.5 BREAKDOWN EVALUATION (ONLY REQUIRED WHEN ANALYZING FOR PESTICIDES) 12
4.6 INITIAL MULTI-POINT CALIBRATION FOR PESTICIDES AND PCBS 12
4.7 LINEAR CALIBRATION 13
4.8 NON-LINEAR CALIBRATION 14
4.9 SECOND SOURCE STANDARDS (INITIAL CALIBRATION VERIFICATION-ICV) 14
4.10 DAILY CALIBRATION CHECK 15
4.11 UPDATING RETENTION TIME WINDOWS 15
4.12 INSTRUMENT BLANK 16
4.13 CONTINUING CALIBRATION VERIFICATION 16
4.14 METHOD BLANKS 17
4.15 BLANK SPIKES (LCS) 17
4.16 MATRIX SPIKE 18
4.17 MATRIX SPIKE DUPLICATE 18
4.18 SURROGATE RECOVERY 19

5 OPERATION PROCEDURES 20

5.1 CHROMATOGRAPHIC CONDITIONS 20
5.2 SAMPLE ANALYSIS 21

6 REPORTS 23

6.1 DATA PACKET ORGANIZATION 23
6.2 QUALITY CONTROL REPORTS 23
6.3 SAMPLE RESULT REPORTS 24
6.4 CONTROL CHART(S) 24

7 REFERENCES **25**

APPENDIX I **26**

5890 STANDARD SOLUTION CONCENTRATIONS 26
6890 STANDARD SOLUTION CONCENTRATIONS 27

APPENDIX II **30**

ANALYSIS SEQUENCE-0.45MM ID 30
ANALYSIS SEQUENCE-0.32MM ID 30

APPENDIX III **31**

DATA PACKET ORDER 31

APPENDIX IV **34**

METHOD 8081B QA REQUIREMENTS AND CORRECTIVE ACTIONS: 34

APPENDIX V **36**

COMPOUND ELUTION ORDER ON DB5 AND DB608 36

APPENDIX VI **37**

COMPOUND ELUTION ORDER ON DBXLB AND DB35MS 37

APPENDIX VII **38**

FLOW CHART 38

APPENDIX VIII **40**

AGENCY REQUIREMENTS FOR SW 8081 40

APPENDIX IX **43**

CONTROL LIMITS 43

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	
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

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.

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.

PQL or Reporting Limit	Practical Quantitation Limit or Reporting Limit- The value used when reporting a non-detect. It may be administratively, empirically or contractually set.
QC check standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC period	Quality control period. An analysis sequence initiated by the analysis of one or more standards, followed by sample extracts/digests, and terminated with a standard analysis. A QC period can be open-ended chronologically, but calibration verification must be documented using the procedures in this SOP.
Reporting Limit (RL)	The reporting limit is defined by the low level standard, also referred to as the practical quantitation limit (PQL).
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values expressed as a percentage. A measure of the similarity of the values one to another.
RT, Retention time	The time (in minutes) at which a target analyte elutes from a chromatography column.
RT window	Retention time window. The +/- value which is applied to the ICV to establish the time range used to make tentative compound identifications.
Sequence	A set of sample extracts/digests and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.
SRM	Standard Reference Material - A material containing known quantities of target analytes in a homogeneous matrix which approximates the matrix of the samples being analyzed. It is used to establish that the analytical process is in control.

2 Equipment List, Maintenance, and Standards

2.1 Chromatographic and Data System

2.1.1 This analysis requires a gas chromatograph with a programmable oven, heated injection port, dual electron-capture detectors, autosampler and an electronic data-acquisition system.

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.

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). High-level INDAB is made at 16 times the PQL concentration. The lower levels are diluted by factors of 2, 4, 5, 8, 16 and 20. 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 seven concentration levels: 1.0 µg/ml, 0.75 µg/ml, 0.5 µg/ml, 0.33 µg/ml, 0.25 µg/ml 0.10 µg/ml, and 0.05 µg/ml. These must be replaced after 6 months or by the expiration date of the material they were made from, 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	0.1	N/A	N/A	N/A
3	0.005-0.01	0.20	0.5	N/A	N/A
4	0.008-0.016	0.25	N/A	N/A	N/A
5	0.02-0.4	0.5	N/A	N/A	N/A
6	0.04-0.8	0.75	N/A	N/A	N/A
7	0.04-0.08				

2.2.4 A performance evaluation mixture (PEM) is prepared from a purchased solution of 4,4'-DDT, and endrin in hexane.

2.2.5 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).

2.2.6 Refer to SOP LTL-1013 (Preparation, Storage, Shelf Life and Traceability Documentation of Standards and Reference Materials) for detailed instructions on standards preparation and storage.

2.2.7 Appendix I details the compounds and concentrations contained in all solutions.

3 Safety precautions and Waste Disposal

3.1 Routine Safety Precautions

3.1.1 All standards and sample extracts should be handled as if they contain hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 The electron-capture detectors used in this analysis contain a radioactive source and should not be opened or otherwise tampered with.

3.2 Waste disposal

3.2.1 Solvent wastes generated in using this procedure (expired standards, old sample extracts, rinsates, etc.) should be emptied into the solvent waste container in the fume hood.

3.2.2 Waste segregation and disposal from the point of collection is further covered in the Laucks SOP on Waste Segregation and Disposal.

4 Calibration and Quality Control

4.1 QA Requirements and Corrective Action

4.1.1 Detailed in the following sections are applicable QA requirements and subsequent corrective actions to be applied to this analysis. A summary of these requirements can be found in Appendix IV.

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP on MDLs, located in the SOP manual. Briefly, it involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method

validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically higher.

4.3.2 The precision of spike recoveries must meet or exceed the criteria tabulated in SW 846.

4.4 Retention Time Windows

4.4.1 Retention time window studies are conducted following the Laucks SOP on Establishing RTWs and the procedure detailed in SW 846, Method 8000B, which defines a window for each single and multi- component analyte based on the retention times taken from three standard injections over a 72 hour period.

4.4.2 The retention time window half-width is set at 3 times the above calculated standard deviation. This operation must be repeated whenever major equipment changes are made, whenever the chromatographic method is significantly modified, or whenever a column is replaced.

4.4.3 The calculated retention time window half widths are typically unrealistic values. Therefore RT windows have been administratively set at the values listed in OLM04.2 for megabore analyses (see table below) and at ±0.03 for capillary analyses. These values are typically wider than 3 times the standard deviation as determined above, but are more realistic.

Analyte	Megabore	Capillary
alpha-BHC	±0.05	±0.03
beta-BHC	±0.05	±0.03
gamma-BHC (Lindane)	±0.05	±0.03
delta-BHC	±0.05	±0.03
Heptachlor	±0.05	±0.03
Aldrin	±0.05	±0.03
alpha-Chlordane	±0.07	±0.03
gamma-Chlordane	±0.07	±0.03
Heptachlor epoxide	±0.07	±0.03
Dieldrin	±0.07	±0.03
Endrin	±0.07	±0.03
Endrin aldehyde	±0.07	±0.03
Endrin ketone	±0.07	±0.03
4,4'-DDD	±0.07	±0.03
4,4'-DDE	±0.07	±0.03
4,4'-DDT	±0.07	±0.03
Endosulfan I	±0.07	±0.03
Endosulfan II	±0.07	±0.03
Endosulfan sulfate	±0.07	±0.03
Methoxychlor	±0.07	±0.03
Tetrachloro-m-xylene	±0.07	±0.03
Decachlorobiphenyl	±0.10	±0.03
Aroclors	±0.07	±0.03
Toxaphene	±0.07	±0.03

4.5 Breakdown Evaluation (only required when analyzing for pesticides)

4.5.1 At the beginning of each analysis sequence a performance evaluation mixture (PEM) must also be analyzed. This is a separate solution containing 4,4'-DDT and endrin, and is examined for the breakdown products of these analytes (4,4'-DDD, 4,4'-DDE, endrin aldehyde, endrin ketone) which indicate the need for GC system maintenance. The percent breakdown must be less than 15% for both Endrin and 4,4'-DDT.

4.6 Initial Multi-Point Calibration for Pesticides and PCBs

4.6.1 SW 846 allows the use of both linear and non-linear models for the calibration data. The option for non-linear calibration may be necessary to achieve low detection limits or to address specific instrumental techniques.

4.6.2 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.

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.

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 ≥ 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.

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 Aroclor 1660, 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:

$$\%D = \frac{C_V - \overline{CF}}{\overline{CF}} \times 100$$

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

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-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.

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:

$$\%\text{RPD} = \frac{|\chi - \delta|}{(\chi + \delta)/2} x100$$

where:

χ = measured concentration for MS sample
δ = 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 indicators of poor method performance than high surrogate recovery since non-GC/MS methods cannot separate co-eluting interferences. Hence corrective action is not required for high surrogate recoveries.

4.18.2.2 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

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 particular pair of columns and detectors. Once optimized, the same conditions must be used for the analysis of all standards, samples, blanks and spikes.

5.2 Sample Analysis

5.2.1 Analysis sequence

5.2.1.1 See Appendix II for a detailed analysis injection sequence.

5.2.2 Compound Identification

5.2.2.1 Compounds are tentatively identified if a peak elutes in the retention time window characteristic of that compound on the column 1. To confirm the presence of that compound in the sample extract, the peak must also elute in its characteristic retention time window on a second column. Retention time windows are established as previously described and the absolute retention times are updated each QC period. Compounds can only be identified if the ICV and CCV criteria previously detailed are strictly adhered to.

5.2.2.2 The experienced analyst's judgment weighs heavily in evaluating chromatograms for compound identification. For instance, the retention times of surrogate compounds may be outside their expected windows due to sample matrix effects. The analyst may

decide to re-adjust the target analyte's retention time windows on an ad hoc basis based on such an observed shift. This can occur only on a sample-specific basis and is used when the analyst examining the data suspects that a retention time shift has occurred. If this is done, it must be fully documented in the case narrative notes.

5.2.2.3 Identification of multicomponent analytes occurs when the retention times and ratios of each of the multicomponent peaks present in the sample match the peaks chosen for the particular multicomponent analyte in the initial calibration. These same conditions must be met on both columns in order to confirm the presence of the multicomponent analyte. The experienced analyst's judgment weighs heavily in evaluating the patterns of multicomponent analytes with regards to weathering and matrix interferences.

5.2.3 Compound Quantitation

Target compound concentrations are calculated using the following equations:

5.2.3.1 Aqueous samples

5.2.3.1.1 The external standard equation, as expressed in SW 846 is:

$$\text{Concentration } (\mu g / L) = \frac{(A_x)(V_t)(D)}{(CF_m)(V_i)(V_s)}$$

where:

A_x = Response for the analyte in the extract, in area or height units.
CF_m = Multi-point average CF
V_i = Volume of extract injected, µL.
D = Dilution factor of extract. The final result of an algebraic multiplication of the ratio of all dilution final volumes to initial volumes. For example, if and extract was diluted 100 µL to 1000 µL and subsequently diluted an additional 100 µL to 1000 µL, the expression would be: (1000/10) * (1000/10) = 100 * 100 = 10,000. If no dilution was made, D = 1.
V_t = Volume of total extract, µL.
V_s = Initial sample size, mL.

The reported concentration for multicomponent analytes calculated is based on an average of the concentrations determined for each of the peaks chosen for calibration.

5.2.3.1.2 To report concentrations in alternate units, apply an appropriate factor:

mg/L = µg/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.

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 lower result is reported in order to employ the conservative approach relative to protection of the environment.

6 Reports

6.1 Data Packet Organization

6.1.1 See Appendix III for a checklist detailing data packet organization.

6.2 Quality Control Reports

6.2.1 All results for quality control tests are entered into the quality control database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and an MS/MSD report.

6.3 Sample Result Reports

6.3.1 Data Qualifying Flags

6.3.1.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the reporting limit indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantification limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
P	When a dual column /dual detector GC technique is employed, this flag indicates that calculated results from the two determinations differ by more than 25%. If the results from one column is significantly higher (25%) the chromatogram is checked for overlapping peaks, or irregular baseline integration. If no anomalies are discovered, the higher result is reported in order to employ the conservative approach relative to protection of the environment.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported should be considered an estimate.
C	The target analyte's presence was confirmed by GC/MS.

6.4 Control Chart(s)

6.4.1.1 The recovery values for 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 .

APPENDIX I

5890 Standard Solution Concentrations, µg/L

Compound	STD1	STD2	STD3	STD4	STD5
alpha-BHC	5.0	10.0	20.0	40.0	80.0
beta-BHC	5.0	10.0	20.0	40.0	80.0
delta-BHC	5.0	10.0	20.0	40.0	80.0
gamma-BHC (Lindane)	5.0	10.0	20.0	40.0	80.0
alpha-Chlordane	5.0	10.0	20.0	40.0	80.0
gamma-Chlordane	5.0	10.0	20.0	40.0	80.0
Heptachlor	5.0	10.0	20.0	40.0	80.0
Aldrin	5.0	10.0	20.0	40.0	80.0
Heptachlor epoxide	5.0	10.0	20.0	40.0	80.0
Endosulfan I	5.0	10.0	20.0	40.0	80.0
Dieldrin	10.0	20.0	40.0	80.0	160
4,4'-DDE	10.0	20.0	40.0	80.0	160
Endrin	10.0	20.0	40.0	80.0	160
Endosulfan II	10.0	20.0	40.0	80.0	160
4,4'-DDD	10.0	20.0	40.0	80.0	160
Endosulfan sulfate	10.0	20.0	40.0	80.0	160
4,4'-DDT	10.0	20.0	40.0	80.0	160
Methoxychlor	50.0	100	200	400	800
Endrin aldehyde	10.0	20.0	40.0	80.0	160
Endrin ketone	10.0	20.0	40.0	80.0	160
Toxaphene			500		
Aroclor-1016	100	250	500	1000	2000
Aroclor-1221			500		
Aroclor-1232			500		
Aroclor-1242			500		
Aroclor-1248			500		
Aroclor-1254			500		
Aroclor-1260	100	250	500	1000	2000
2,4,5,6-tetrachloro-m-xylene	5.0	10.0	20.0	40.0	80.0
Decachlorobiphenyl	10.0	20.0	40.0	80.0	160

6890 Standard Solution Concentrations, µg/L

Compound	STD1	STD2	STD3	STD4	STD5	STD6	STD7
alpha-BHC	4.0	5.0	10.0	16.0	20.0	40.0	80.0
beta-BHC	4.0	5.0	10.0	16.0	20.0	40.0	80.0
delta-BHC	4.0	5.0	10.0	16.0	20.0	40.0	80.0
gamma-BHC (Lindane)	4.0	5.0	10.0	16.0	20.0	40.0	80.0
alpha-Chlordane	4.0	5.0	10.0	16.0	20.0	40.0	80.0
gamma-Chlordane	4.0	5.0	10.0	16.0	20.0	40.0	80.0
Heptachlor	4.0	5.0	10.0	16.0	20.0	40.0	80.0
Aldrin	4.0	5.0	10.0	16.0	20.0	40.0	80.0
Heptachlor epoxide	4.0	5.0	10.0	16.0	20.0	40.0	80.0
Endosulfan I	4.0	5.0	10.0	16.0	20.0	40.0	80.0
Dieldrin	8.0	10.0	20.0	32.0	40.0	80.0	160
4,4'-DDE	8.0	10.0	20.0	32.0	40.0	80.0	160
Endrin	8.0	10.0	20.0	32.0	40.0	80.0	160
Endosulfan II	8.0	10.0	20.0	32.0	40.0	80.0	160
4,4'-DDD	8.0	10.0	20.0	32.0	40.0	80.0	160
Endosulfan sulfate	8.0	10.0	20.0	32.0	40.0	80.0	160
4,4'-DDT	8.0	10.0	20.0	32.0	40.0	80.0	160
Methoxychlor	40.0	50.0	100	160	200	400	800
Endrin aldehyde	8.0	10.0	20.0	32.0	40.0	80.0	160
Endrin ketone	8.0	10.0	20.0	32.0	40.0	80.0	160
Toxaphene				250			
Aroclor-1016	50	100	250	330	500	750	
Aroclor-1221			250				
Aroclor-1232			250				
Aroclor-1242			250				
Aroclor-1248			250				
Aroclor-1254			250				
Aroclor-1260	50	100	250	330	500	750	
2,4,5,6-tetrachloro-m-xylene	4.0	5.0	10.0	16.0	20.0	40.0	80.0
Decachlorobiphenyl	8.0	10.0	20.0	32.0	40.0	80.0	160

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.

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

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)

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

APPENDIX III, continued

Other Standards Used to Support Sample Data and Instrument Blanks

V. Raw QC Data:

_____ Method Blank
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Blank Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

_____ Matrix Spike Duplicate
_____ Chromatograms, column 1
_____ Chromatograms, column 2
_____ Chromatographic Report, column 1
_____ Chromatographic Report, column 2

APPENDIX III, continued

V. Bench Sheets

_____ 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.

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

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

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

APPENDIX VI

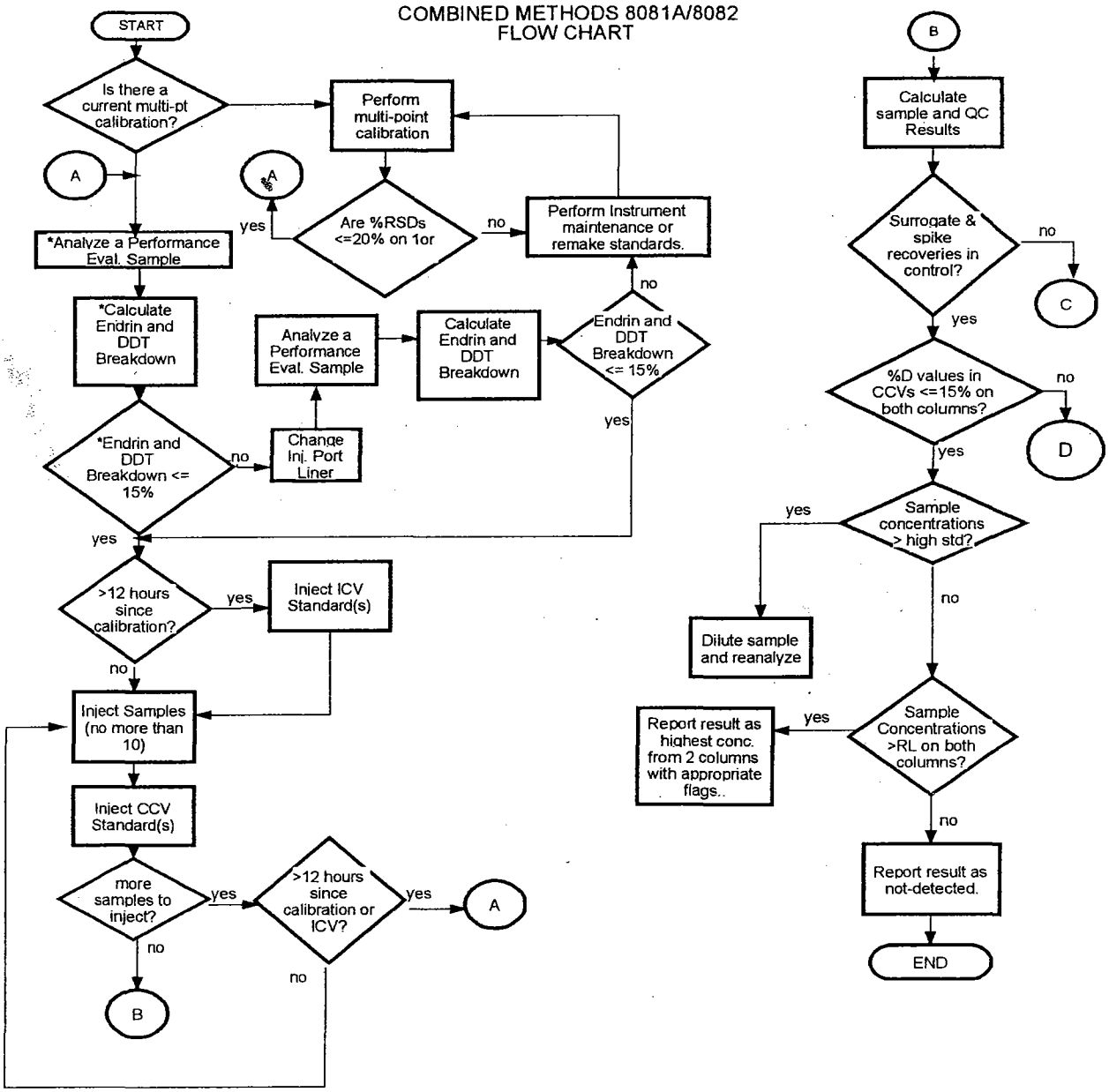
Compound Elution order on DBXLB and DB35ms Capillary (0.32 mm ID) Columns

DBXLB	DB35ms
Tetrachloro-m-xylene	Tetrachloro-m-xylene
alpha-BHC	alpha-BHC
gamma-BHC (Lindane)	gamma-BHC (Lindane)
beta-BHC	beta-BHC
delta-BHC	Heptachlor
Heptachlor	delta-BHC
Aldrin	Aldrin
Heptachlor epoxide	Heptachlor epoxide
gamma-Chlordane	gamma-Chlordane
alpha-Chlordane	alpha-Chlordane
Endosulfan I	Endosulfan I
4,4'-DDE	4,4'-DDE
Dieldrin	Dieldrin
Endrin	Endrin
4,4'-DDD	4,4'-DDD
Endosulfan II	Endosulfan II
Endrin aldehyde	4,4'-DDT
4,4'-DDT	Endrin aldehyde
Endosulfan sulfate	Endosulfan sulfate
Methoxychlor	Methoxychlor
Endrin ketone	Endrin ketone
Decachlorobiphenyl	Decachlorobiphenyl

APPENDIX VII

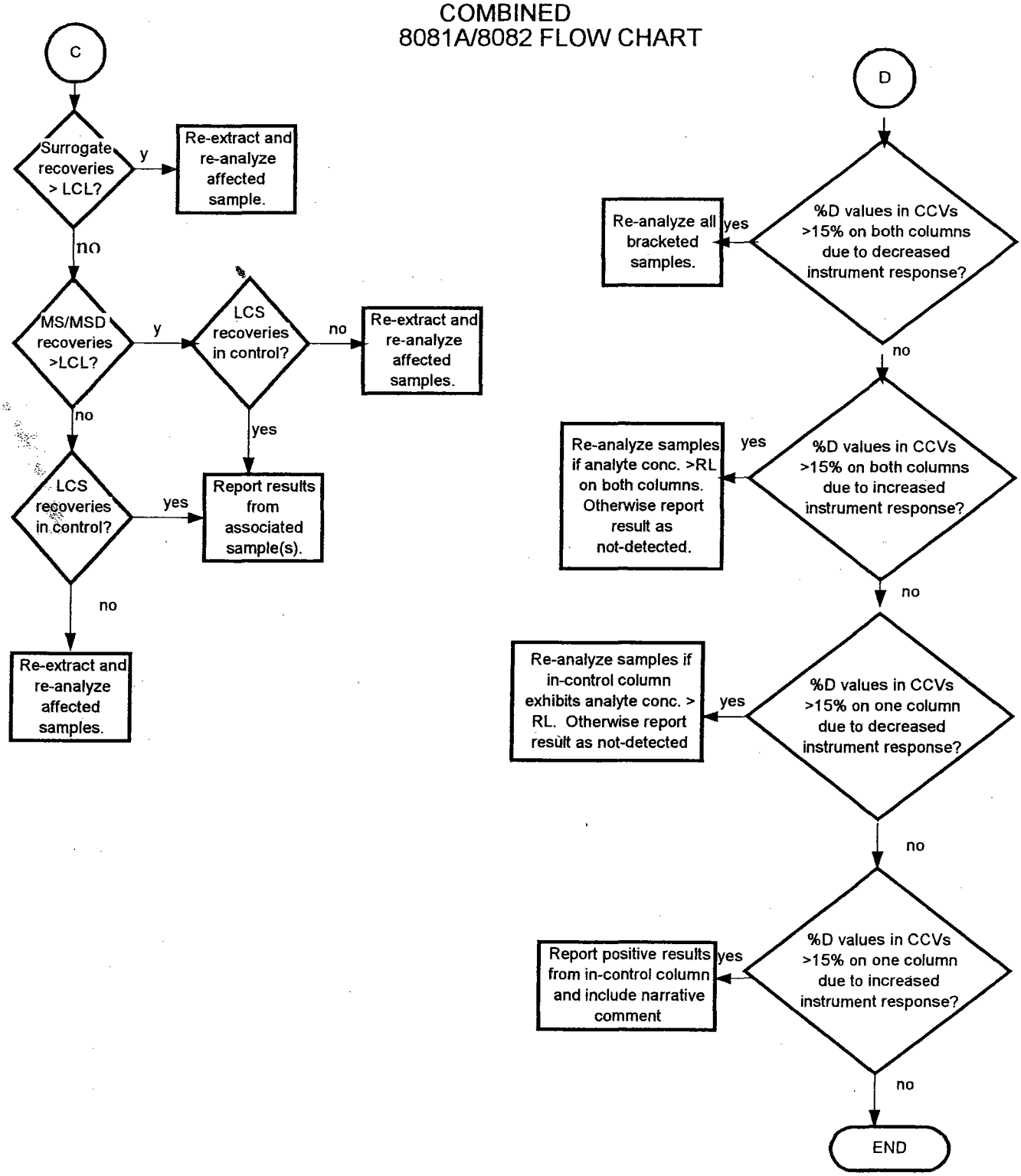
Flow Chart

COMBINED METHODS 8081A/8082
FLOW CHART



*These steps can be eliminated if analyzing for SW8082 compounds only

COMBINED 8081A/8082 FLOW CHART



Appendix VIII

Agency Requirements for SW 8081

QC Check	DoD	AFCEE	USACE	SW 846
	Acceptance Criteria	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: Averaged - Grand mean RSD ≤ 20%	Option 1: RSD ≤ 20%	Option 1: Averaged - RSD ≤ 20%
	Option 2: Linear - r ≥ 0.995	Option 2: Linear - r ≥ 0.995	Option 2: Linear - 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 regression: coefficient of determination r^2 ≥ 0.99	Option 3: Non-linear regression: 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 ± 15%	within ± 15%	

QC Check	DoD	AFCEE	USACE	SW 846
	Acceptance Criteria	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
Continuing Calibration Verification	Within ± 20%.	Within ± 15%.	Within ± 15%.	Within ± 15%.
	Every 10 field samples, beginning and end of analytical run	Every 10 samples, beginning and end of analytical sequence		Beginning and end of each 12 hour analytical shift
				Required after every 20, recommended after 10
Method Blank	No analytes ≥ 1/2 RL. No common laboratory contaminant analytes ≥ RL	No analytes ≥ RL	Analytes < 1/2 MRL	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	See appropriate AFCEE table	50%-130% recovery	Lab-derived

QC Check	DoD	AFCEE	USACE	SW 846
	Acceptance Criteria	Acceptance Criteria	Acceptance Criteria	Acceptance Criteria
	not listed			
Matrix Spike	Same as LCS	Same as LCS	40%-140% recovery	Lab-derived
Matrix Spike Duplicate	RPD ≤ 30%	Water: RPD ≤ 30%	RPD ≤ 50%	Lab-derived
		Soil: RPD ≤ 50%		
Surrogate Spike	Same as LCS	Same as LCS	Blank Spike: 50%-130% recovery	Lab-derived
			Matrix Spike: 40%-140% recovery	
Confirmation of positive results	RPD ≤ 40	RPD ≤ 40%, J flag if greater	RPD ≤ 40%	RPD ≤ 40%
Results reported between MDL and RL	Apply J flags	Apply F flags		
Allowable Sporadic ME/Failures	11 - 30 analytes – 1 1 – 10 analytes: 0			

Appendix IX

Control Limits

Water

Target Analyte	RL	MDL	Blank	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	

Soil

Target Analyte	RL	MDL	Blank	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	

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

LAUCKS TESTING LABORATORIES INC.
Seattle, Washington

SOP #:LTL-8265

Title: **Determination of Volatile Organic Compounds by Method SW 8260B**

Revision history:

Number	Date
0	01/29/98
1	06/17/98
2	04/20/00
3	10/2/01
4	03/08/02
5	05/30/02
6	11/05/02
7	11/22/02
8	04/30/04
9	08/15/05

Revised by: Joyell Dunay, GC/MS Volatiles Analyst Date: 8/16/05

Approved by: Harry Romberg, QA Officer Date: 8/16/05

Approved by: Kathy Kreps, Laboratory Director Date: 8/18/05

Controlled Document

No. 20 Assigned to: TETRA

TABLE OF CONTENTS

1. INTRODUCTION AND SCOPE 4

1.1 METHOD DESCRIPTION 4
1.2 METHOD DEVIATIONS 4
1.3 INTERFERENCES 4
1.4 SAMPLE COLLECTION, SAMPLE STORAGE, HOLDING TIMES 5
1.5 DEFINITION OF TERMS 7

2. EQUIPMENT LIST AND STANDARDS 9

2.1 CHROMATOGRAPHIC SYSTEM 9
2.2 STANDARDS 10

3. SAFETY PRECAUTIONS & WASTE DISPOSAL 15

3.1 ROUTINE SAFETY PRECAUTIONS 15
3.2 WASTE DISPOSAL 16

4. OPERATION PROCEDURES 16

4.1 ANALYTICAL CONDITIONS 16
4.2 METHOD DETECTION LIMIT STUDY 17
4.3 METHOD VALIDATION 17
4.4 INSTRUMENT TUNING 17
4.5 INITIAL MULTI-POINT CALIBRATION 19
4.6 CONTINUING CALIBRATION VERIFICATION 20
4.7 INSTRUMENT BLANK 20
4.8 METHOD BLANK 21
4.9 SAMPLE ANALYSIS 21
4.10 ANALYSIS SEQUENCE 21
4.11 SAMPLE PREPARATION 21
4.12 QUALITATIVE IDENTIFICATION 22
4.13 MANUAL PEAK INTEGRATIONS 23
4.14 COMPOUND QUANTIFICATION 24

5. REPORTS 26

5.1 DATA PACKET ORGANIZATION 26
5.2 QUALITY CONTROL REPORTS 26
5.3 CONTROL LIMITS 26
5.4 DATA QUALIFYING FLAGS 26

6. QUALITY CONTROL 27

6.1 INITIAL CALIBRATION 27
6.2 CONTINUING CALIBRATION VERIFICATION 29
6.3 INSTRUMENT BLANK 31
6.4 METHOD BLANKS 31
6.5 BLANK SPIKES 33
6.6 MATRIX SPIKE 34
6.7 MATRIX SPIKE DUPLICATE 35

Controlled Document
Assigned to: _____ No. _____

6.8 SURROGATE RECOVERY 35
7. REFERENCES 37
APPENDIX I 38
INITIAL CALIBRATION CONCENTRATIONS FOR WATERS (µG/L) AND MEDIUM SOILS (µG/KG) 38
APPENDIX II 42
DATA PACKET SEQUENCE 42
APPENDIX III 43
DETERMINATION OF VINYL CHLORIDE BY SELECTED ION MONITORING (SIM) 43
APPENDIX IV 45
APPENDIX IX COMPOUNDS AND THEIR INITIAL CONCENTRATIONS 45
APPENDIX V 46
METHOD 8260 REQUIREMENTS AND CORRECTIVE ACTIONS 46
APPENDIX VI 49
METHOD 8260B FLOW CHART 49
APPENDIX VII 50
METHOD 8260 TARGET ANALYTES 50
APPENDIX VIII 51
METHOD 8260 OPTIONAL ANALYTES FOR LOW LEVEL WATER ANALYSES 51
APPENDIX IX 52
ADDITIONAL APPENDIX IX COMPOUNDS 52
APPENDIX X 53
TARGET ANALYTES AND THEIR ASSOCIATED INTERNAL STANDARDS 53
APPENDIX XI 55
TARGET ANALYTES AND THEIR QUANTITATION IONS 55
APPENDIX XII 57
TARGET ANALYTES, THEIR ELUTION ORDER AND STANDARD MIXES 57
APPENDIX XIII 61
REPORTING LIMITS, MDLS, AND CONTROL LIMITS 61
APPENDIX XIV 68
AGENCY REQUIREMENTS 68

1. Introduction and Scope

1.1 Method Description

Method 8260 is used in the determination of volatile organic compounds in soil, sediment, aqueous, and other matrices. This SOP addresses the determination of volatile organics in different matrices, which include medium level soils as well as low level water samples. In addition, the instrument acquisition parameters have been added for the analysis of vinyl chloride by Selected Ion Monitoring (SIM) in Appendix III, and Appendix IV contains a tabular list of the non-routine Appendix IX analytes and their respective calibration ranges.

1.1.1 Because this method employs sample introduction via a purge and trap sample concentrator, it is applicable to volatile compounds that have boiling points below 200°C and that are insoluble or only slightly soluble in water. Compounds that are more soluble in water have elevated detection limits due to their decreased purging efficiency. These include, but are not limited to ketones, nitriles, acetates, acrylates and ethers.

1.1.2 This method is restricted to use by, or under the supervision of analysts experienced in the use of purge and trap systems, gas chromatography/mass spectroscopy and in the interpretation of chromatograms and mass spectral data. Each analyst performing this method must have demonstrated the ability to perform the described chromatographic analysis and data interpretation.

1.2 Method Deviations

1.2.1 Detailed below are any deviations from the published version of method SW 8260B. All deviations are followed as the standard operation procedure by Laucks Testing Labs.

1) Laucks uses 1/2 the reporting limit (low level standard) for method blank criteria instead of method detection limits as referenced in SW 846. Refer to Section 6.4 for detailed method blank criteria.

2) The retention time window used for internal standards in all analyses is +/- 0.50 minutes relative to the daily CCV standard in lieu of using 0.06 RRT units.

1.3 Interferences

1.3.1 Problems have been associated with the following compounds analyzed by this method.

1.3.1.1 Chloromethane is the most likely compound to be lost if the purge flow is too fast.

1.3.1.2 Bromoform is one of the compounds to most likely to be purged very poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.

1.3.1.3 Tetrachloroethane and 1,1-dichloroethane are degraded by contaminated transfer lines in the purge-and-trap system and/or active sites in trapping materials.

1.4 Sample Collection, Sample Storage, Holding Times

1.4.1 Waters

1.4.1.1 Samples are collected with zero headspace in 40-mL glass containers containing preservative (either HCl, or solid $NaHSO_4$) and sealed with Teflon-lined caps.

1.4.1.2 The water samples are preserved to a pH of ≤2, and all samples are stored at 4°C±2°.

1.4.1.3 The holding time to analysis is 14 days from date of collection. The pH values for the water samples are measured subsequent to sample analysis. The pH values, when not preserved to a pH ≤2 are noted in the instrument logbook and client report. The project manager will also be notified **as soon as** any out of control pH is discovered.

1.4.2 Soils

1.4.2.1 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that low level soils be collected in duplicate using 5 gram and/or 1 gram aliquots which are to be weighed out in the field. The soil samples are to be collected in a 40-mL vial containing water, sodium bisulfate, and a stir bar. 0.2 grams of sodium bisulfate are added for each 1 gram of soil (see section 1.4.2.2 for issues associated with this method). Therefore, when collecting 5 grams of sample, the VOA vial should contain 1 gram of sodium bisulfate. The sodium bisulfate is added in order to ensure a sample pH of ≤2. The vials used for low-level soils should have the tare weight recorded on the label and sealed with septum-lined screw caps.

1.4.2.2 An in-depth study of the different sampling methods used for Method SW 846 5035 was performed by the IT Corporation and presented to the EPA at it's annual conference in May 2001.

1.4.2.3 Problems associated with sodium bisulfate were confirmed, including styrene degradation, acetone formation, and effervescence in some samples. Dangerous pressure and high methyl acetate results were also noted with sodium bisulfate. EPA allows it only in limited circumstances.

1.4.2.4 Another option is the use of the EnCore™ sampler, the Purge-and-Trap Sampler™, and a cut plastic syringe to transfer the sample to the soil vial. If the EnCore™ sampler is used in the field, the samples may be stored for up to 48 hours. At the time of this writing, SW-846 method 5035 acknowledges that this storage device has not been thoroughly evaluated and suggests that samples collected in this device should be transferred to the soil sample vials (and preserved) as soon as possible, or analyzed within 48 hours. However, studies from Hewitt and Sorini (see reference section 7.0) have shown that freezing EnCores will result in very little loss of volatiles and should have an extended holding time of 14 days. Based on these studies, Laucks also feels freezing EnCores is an appropriate preservation method. However, if required by the client, and for USACE samples, Laucks will extrude 3 EnCore™ samples into vials (and freeze) at the time of receipt. Other client-specified methods of preservation may also be used. Additional samples will be preserved (by freezing) for designated QC samples. The holding time for soils is 14 days from the date of collection.

1.4.2.5 At the time of this writing, the 3rd update of the 3rd edition to SW 846 requires that high level soils be collected using one of the following options:

1.4.2.5.1 Option 1: Samples will be collected in duplicate using 5 gram and/or 1 gram aliquots which are to be weighed out in the field. The soil samples are to be collected in a 40-mL vial containing methanol. The vials used for high level soils should have the tare weight recorded on the label and sealed with septum-lined screw caps.

1.4.2.5.2 Option 2: Samples will be collected using an EnCore type sampler or equivalent, and immediately preserved at the laboratory by adding methanol. (Based on the studies above, Laucks may also freeze the EnCores and add methanol at the time of analysis.)

1.4.2.5.3 Option 3: Samples will be collected in vials with zero headspace.

1.4.2.6 Per client request Laucks will continue to accept unpreserved soil samples for low or high level analyses. The samples must be collected in 2- or 4-oz glass containers and sealed with septum-lined caps. These samples will be introduced by method 5035A and analyzed according to this SOP.

1.4.2.7 The holding time to analysis is 14 days from date of collection.

1.5 Definition of Terms

1.5.1 This section defines terms and acronyms as they are used in this SOP. Other terms, such as MS/MSD or method blank, are not defined here since it is assumed that the user of this SOP already understands their more general meaning.

Blank Spike A background free matrix (DIW for water, clean sand for soils/sediments) to which known amounts or target analytes and surrogates are added each time samples are analyzed. In the context of this SOP, a blank spike is the same as a QC check standard. See also QC check standard.

CCC Calibration check compound. There are 6 analytes that must not exceed the maximum %RSD of 30% in the initial calibration and a %D of 20% in the continuing calibration standard to validate linearity. These analytes are 1,1-dichloroethene, chloroform, 1,2-dichloropropane, toluene, ethylbenzene and vinyl chloride.

CCV Continuing calibration verification. This is a standard analyzed at some prescribed frequency during the analysis sequence to determine whether the instrument has remained in calibration.

CLP Contract Laboratory Program. The USEPA program that contracts with laboratories to provide laboratory services. The term has come to mean a much broader set of methods and deliverables. In the context of this SOP, CLP means procedures or operations which are detailed in the CLP contract and which are extended to a broader working definition.

IBLK Instrument blank. An instrument blank is PFW containing the method surrogates and is introduced into the instrument to monitor for carry over between sample analyses.

ICV Initial continuing verification. A calibration standard obtained from a different manufacturer than the standards used for the initial and continuing calibrations that is analyzed after a compliant initial calibration.

IDL Instrument detection limit. The lowest concentration of a target analyte that will yield a signal:noise ratio of at least 3x. Used as a starting point for selecting MDL study spiking levels.

MDL	Method detection limit. The lowest concentration in a sample which will yield a positive result that is greater than zero at a known level of confidence. MDLs are empirically determined by Laucks.
PFW	Purge-free water. Laboratory deionized water that is boiled for 30 minutes prior to use. The systems used to provide deionized water at Laucks all contain carbon-polishing filters that are capable of providing purge-free water for use as reagent water.
QC Check Standard	Quality control check standard. Referred to in this SOP as a blank spike. A QC check standard is a requirement of SW 846 method 8000 and is used to determine whether the analytical system is in control if MS/MSD recoveries are out of control. See also blank spike.
QC Period	Quality control period. An analysis sequence initiated by the injection of BFB, followed by the standard. A QC period is terminated after 12 hours from the injection of BFB.
Reporting Limit (RL)	The reporting limit is defined by the low level standard, also referred to as the practical quantitation limit (PQL).
RF	Response factor. A measure of the response of an analyte compared to its internal standard response. Response factors are determined by standard analysis and are used in the calculation of concentrations of analytes in samples.
RRT	Relative retention time. A measure of the retention time of an analyte compared to the retention time of its internal standard.
RSD or %RSD	Relative standard deviation or percent relative standard deviation. The ratio of the standard deviation of a set of values to the mean of the set of values. A measure of the similarity of the values one to another.
RT	Retention time. The time (in minutes) at which a target analyte elutes from the GC column.

Sequence A set of samples and standard solutions introduced into an instrument in a chronologically continuous group. See also QC period.

SPCC System performance check compound. Specified compounds in which the minimum RFs must be met in order to demonstrate that the initial and continuing calibration standards are in control. These compounds are chloromethane, 1,1-dichloroethane, bromoform, chlorobenzene, and 1,1,2,2-tetrachloroethane.

2. Equipment List and Standards

2.1 Chromatographic System

2.1.1 Gas Chromatograph: Hewlett Packard 5890 I or 6890 employing a low-dead-volume interface from the sample concentrator to the GC injection port.

2.1.2 Carrier Gas: Helium 99.995% (high purity grade) or better.

2.1.3 Column: 20m x 0.18mm x 1.0 µm, or 75 m x 0.45 x 2.55 µm film thickness, fused silica capillary column (J&W DB-624 or Zebron ZB-624 or equivalent).

2.1.4 Purge and Trap: OI Analytical 4560 or Eclipse sample concentrators or equivalent.

2.1.5 Autosampler: Dynatech Precision Sampling PTA-30W/S, Varian Archon or equivalent.

2.1.6 GC/MS Interface: Hewlett Packard 59913A jet separator assembly, split/splitless or equivalent.

2.1.7 Mass Spectrometer: Hewlett Packard 5970B, 5973 or equivalent.

2.1.8 Data System: HP Chemstation Revisions B.01.00, D.00.01 and D.01.00 Sp1, Environmental Processing, and EnviroForms.

2.1.9 Trap: OI Corporation, Supelco, or equivalent. Examples are listed below:

1) OI #10 Trap. Tenax, silica gel, and charcoal. OI Pt. No.:228122.

2) VOCARB 3000. Supelco Pt No.: 24940-U.

2.1.10 Miscellaneous: Assorted gas-tight calibrated syringes, pipettes, 40 mL vials, caps, septa, sea sand, purge and trap grade methanol, ferrules, pH strips, purge-free DIW, volumetrics, laboratory oven capable of heating glassware up to 100°C, and assorted supplies.

2.2 Standards

2.2.1 Overview of Volatile Standards Preparation

2.2.1.1 All standards are made using a high purity purge and trap grade methanol. Every lot number of methanol is analyzed prior to use to ensure that it is free of volatile organic contaminants. A record of analysis of an aliquot from each batch is stored for a minimum of one year.

2.2.1.2 The LIMS standard logbook is employed to document all volatile standards. All standards are assigned a unique identifier to enable cross-referencing of each individual standard back to the supplier's lot number. In addition, all standards are labeled with the standard concentration, the solvent, date prepared, expiration date, analyst's initials, and the standard reference number. Refer to Laucks' SOP LTL-1013 on the traceability, documentation, and preparation of standards.

2.2.1.3 All standards must be identified and quantitated prior to use in the laboratory. This is performed by purchasing certified standard solutions from a commercial supplier that is recognized by the EPA and which are traceable to NIST. In addition, standards are monitored frequently by comparison to the initial calibration curve. Fresh standards should be prepared if the calibration standard(s) fail to meet the criteria specified in SW 846. Standards for gases need to be replaced after one week, or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. Standards for the non-gases need to be replaced after six months or as recommended by the standard manufacturer, unless the acceptability of the standard can be documented. If the standards meet the criteria for the calibration standard(s) as specified in SW 846, they are considered acceptable.

2.2.1.4 All standard solutions are stored in the VOA freezer at -10°C to -20°C. The stock and secondary dilution standards are stored in flame-sealed amber ampules (with the exception of the internal and surrogate standards). The secondary dilution standards are checked for degradation or evaporation prior to preparing calibration standards. Secondary dilution standards should be replaced after one week, unless the acceptability of the standard can be documented. Pre-mixed certified solutions are stored according to the manufacturers' documented holding time and storage temperature recommendations. Prior to daily and initial calibration standard preparation, the

standard solutions are removed from the freezer and allowed to warm to room temperature.

2.2.1.5 Refer to Appendix I for a tabular listing of all standard solutions and their concentrations. The additional Appendix IX and California oxygenate compounds (which are only analyzed upon request) and their standard concentrations are tabulated in Appendix IV.

2.2.2 Preparation of Internal Standards and Surrogate Standards

2.2.2.1 If starting with a neat material, a measured amount of the neat chemical is placed in a glass volumetric and diluted to volume with methanol, then stoppered. 125 mg of neat material is diluted into 25 mL of methanol. This yields an intermediate solution of 5000 µg/mL.

2.2.2.2 Aliquots of the intermediate solutions are taken from the individual internal and surrogate standards and then combined in a volumetric flask and diluted with methanol to yield a working standard which contains all compounds at a concentration of 250 µg/mL each. This is accomplished by using a gas-tight syringe to transfer each aliquot of 2.50 mL into 50 mL of methanol. A 1 µL aliquot of this solution is injected via the autosampler into the sample, resulting in an internal and surrogate standard concentration of 50 µg/L.

Internal Standards	Surrogate Standards
Fluorobenzene	1,2-Dichloroethane-d4
Chlorobenzene-d5	Toluene-d8
1,4-Dichlorobenzene-d4	p-Bromofluorobenzene
	Dibromofluoromethane

2.2.3 Preparation of Spike Solution

2.2.3.1 Certified spiking solutions are purchased pre-mixed from commercial suppliers at various concentrations. (See table below.) The solutions are then diluted in methanol to a concentration of 200 µg/mL. Refer to Section 6 for additional information on spiking analytes.

Spike Solution	Source*	Stock Number*	Concentration (µg/mL)	Working Solution (µg/mL)
MTBE	Supelco	48483	2000	200
Gases	Supelco	48799-U	2000	200
Carbon disulfide	Supelco	40363	5000	200
TCL Mix#1	Supelco	48949	2000	200
502.2 Megamix	Restek	30431	2000	200

* or equivalent

2.2.3.2 A 12.5 µL aliquot of this solution is spiked into 50 mL of sample or PFW (blank spike) in a volumetric flask, resulting in a spiked concentration of 50 µg/L for each analyte. This solution is transferred to a 40mL VOA vial (see specific volumes below) and placed onto the autosampler for analysis along with the samples. The following chart summarizes the preparation method for each matrix. (See Appendix XIII for laboratory control limits on the spike recoveries.)

Working Concentration	Aliquot	Final Volume (MS/MSD* use sample, blank spikes use PFW)	Concentration and volume used for analysis
Soils (5g sand)			
200 µg/mL	12.5 µL	50 mL sample or PFW	Pour 5mL of 50 µg/L solution with 5g of sand in 40mL vial
Medium Level Soils			
200 µg/mL	12.5 µL	(Add 1mL of MeOH to blank spikes) fill to 50mL sample or PFW	Fill 50 µg/L solution into 40mL vial
5 mL Water			
200 µg/mL	12.5 µL	50 mL sample or PFW	Transfer 50 µg/L solution to 40mL vial

*For MS/MSD samples all volumes may be doubled to produce 2 vials from the same solution.

2.2.4 Preparation of p-Bromofluorobenzene (BFB) Solution

2.2.4.1 The preparation of BFB is similar to that of the internal standards and surrogate standards. Refer to Section 2.2.2 for details.

2.2.4.2 A solution of 25 ng/mL is prepared and stored in flame-sealed amber ampules.

2.2.5 Preparation of Initial Calibration Standards

2.2.5.1 Certified sets of stock solutions, which contain all target analytes, are purchased pre-mixed from a commercial supplier at a concentration of 2000 µg/mL each. If additional target analytes are requested, these analytes may be ordered separately, either in solution or as a neat material.

Solution	**Source***	**Catalog Number***
APIX (ketones)	Crescent	CC2239.10
APIX	Crescent	CC2464.10
524.2 Vol. Gases	Crescent	CC2006.10
8260 Volatiles	Crescent	CCS-743
Calif. Oxygenates	Restek	30465
Chloroprene	Restek	30238
Ethyl bromide	ChemService	O-640
Acrolein/Acrylonitrile	Crescent	CC2098
1-Chlorohexane	ChemService	O-624
* or equivalent		

2.2.5.2 These solutions are then diluted in methanol to generate standards at concentrations of 200 µg/mL. This solution is stored in flame-sealed amber ampules.

2.2.5.3 Standards are stored in the VOA freezer at -10°C to -20°C. They are removed prior to making up the initial calibration standards and are allowed to warm to room temperature.

2.2.5.4 The initial calibration standards range from 1µg/L to 200µg/L (the low level Std. is 0.3 µg/L for AFCEE work) for low level waters. Soils range from 3 µg/L to 200 µg/L (the low level Std. is 2.0 µg/L for AFCEE work) for all target analytes with the exception of ketones, m,p-xylenes, and various Appendix IX analytes. Please refer to Appendices I and IV for tabular listings of analytes and calibration concentrations. The 5-mL water calibration standards are also employed in the analysis of medium or high level soils, since the aqueous calibration standards are purged at ambient temperature. Analysis of the solid matrices in the low-level format requires a purge temperature of 40° C.

2.2.5.5 The standards are prepared by taking aliquots of the solutions and diluting them into a volumetric flask containing PFW. A 40 mL aliquot is immediately poured into a VOA vial (or a 5 mL aliquot for the low-level soils is placed in a soil vessel) and placed on the autosampler. Each standard concentration is prepared just prior to analysis and any remaining standard solution is disposed of once the analytical run is initiated.

Low Level Soils

Calibration Level	Initial Volume	Final Volume	Final Concentration
VSTD002*	1.0 µL STD	100 mL	2 µg/L
VSTD003	1.5 µL STD	100 mL	3 µg/L
VSTD010	5 µL STD	100 mL	10 µg/L
VSTD020	5 µL STD	50 mL	20 µg/L
VSTD050	12.5 µL STD	50 mL	50 µg/L
VSTD100	25 µL STD	50 mL	100 µg/L
VSTD200	50 µL STD	50 mL	200 µg/L
ICV	12.5 µL ICV	50 mL	50 µg/L
Blank Spikes, MS/MSD	12.5 µL MS	50 mL	50 µg/L

5mL Purge, Low Level Waters and Medium level soils

Calibration Level	Initial Volume	Final Volume	Final Concentration
VSTD0.5*	0.3 µL STD	200 mL	0.3 µg/L
VSTD001	1 µL STD	200 mL	1 µg/L
VSTD005	2.5 µL STD	100 mL	5 µg/L
VSTD010	5 µL STD	100 mL	10 µg/L
VSTD050	12.5 µL STD	50 mL	50 µg/L
VSTD100	25 µL STD	50 mL	100 µg/L
VSTD200	50 µL STD	50 mL	200 µg/L
ICV	12.5 µL ICV	50 mL	50 µg/L
Blank Spikes, MS/MSD	12.5 µL MS	50 mL	50 µg/L

* non-routine (can not meet on all instruments)

2.2.6 Preparation of a Second Source Standard

2.2.6.1 The concentration for the second source standard is 50 µg/L for low and medium level soils and 5 mL waters. This standard is always prepared following an initial calibration to verify standard integrity.

2.2.7 Preparation of a Daily Calibration Standard

2.2.7.1 The concentration of the continuing calibration standard at a level of 50 µg/L for low and medium level soils and 5 mL waters, is also the mid-point of the initial calibration curve. This standard is always prepared just prior to analysis by transferring an aliquot of each of the 200 µg/mL multi-component working solutions into a volumetric containing PFW.

3. Safety precautions & Waste disposal

3.1 Routine Safety Precautions

3.1.1 All standards and samples should be handled as if they are hazardous substances.

3.1.2 Refer to the instrument manufacturer's manual for routine instrument precautions.

3.1.3 Routine precautions include an awareness of the moving parts on the instrument you're using. These parts are often charged with power from an electrical component or with high-pressure gas and have the potential to do harm if not used properly.

3.1.4 Electrical shock - All instruments present the possibility of electrical shock. The operator should take all precautions including ensuring that all instruments are operated with fully grounded power outlets, turning off the instrument and disconnecting the instrument from the electrical power supply before working on any electrical components, etc.

3.1.5 Methanol is purchased in small quantities and kept inside the volatile laboratory to avoid cross contamination from other solvents.

3.1.6 Many analytes are known or suspected carcinogens. Analysts should take the proper precautions such as wearing gloves when working with suspect samples or high level standards or solvents. Additionally, a respirator should be worn and a fume hood utilized for extremely hazardous compounds.

3.2 Waste disposal

3.2.1 Waste solvents and expired standards are disposed of in the appropriate waste solvent container located in the prep area under the hood.

3.2.2 Solid sample matrices are disposed of in appropriate drums labeled for this specific purpose where they are routinely picked up by an approved agency for final disposal.

4. Operation procedures

4.1 Analytical Conditions

GC Parameters*	5970	5973
Injection Port Temperature	: 200°C	200°C
GC/MS Interface Temperature	: 250°C	250°C
Initial GC Temperature	: 35°C	40 °C
Initial Hold Time	: 4 min	4 min
Ramp Rate	: 4°C/min	10-15°C/min
Final Temperature	: 180°C	200°C
Final Time	: 4 min	4 min
Carrier Gas Flow	: 15 mL/min	
Column Head Pressure	: 9 psi	

Purge and Trap Parameters*	4560	Eclipse
Purge Time	:11 min	11 min
Desorb Time	:0.8 min	0.8 min
Bake Time	:5-8 min	5-8 min
Desorb Preheat Temperature	:150°C	180°C
Desorb Temperature	:200°C	200 °C
Purge Flow	:40 mL/min	40 mL/min
Purge Temperature	:Ambient (40°C for soils)	Ambient (40°C for soils)

Mass Spectrometer Parameters*	All
Electron Energy	: 70 volts
Mass Range	: 35 to 300 AMU
Scan Time	: 1.4 sec/scan
Scan Start Time	: 0.1 min

* parameters may vary slightly by instrument

4.2 Method Detection Limit Study

4.2.1 Prior to the analysis of any samples, it is necessary to establish method detection limits. This procedure is fully described in the Laucks SOP for the determination of method detection limits. It involves the analysis of 7 replicate samples spiked at a concentration near the anticipated method detection limit. A Student's T-test is then applied to these measured values to calculate the MDL.

4.2.2 Additional MDL studies are performed on an ongoing basis. This procedure is fully described in LTL-1011, Procedure for the Determination and Reporting of Detection Limits, Reporting Limits, Precision and Accuracy Studies, and Control Limits. The MDL values at the time this SOP was written are located Appendix XIII. Current MDL values are found in the LIMS MDL database and associated raw data are stored in department files

4.3 Method Validation

4.3.1 Prior to the analysis of any samples, it is necessary to validate the method. A method validation study is performed in a similar manner to an MDL study with the exception that a minimum of 4 replicates are required and the concentration levels are typically 10-50 times the MDL. In the absence of criteria for the initial demonstration of proficiency, the limits of 70%- 130% can be used.

4.4 Instrument Tuning

4.4.1 Prior to the analysis of any samples, blanks, or calibration standards, the instrument must demonstrate mass calibration and resolution by meeting the established tuning criteria for BFB.

4.4.2 FC-43 (PFTBA) is used as a mass calibrant. The following ratios are suggested in order to meet the tuning criteria for BFB:

Ion	% of ion 69	Isotope	Isotope Abundances
69	100%	70	0.5-1.5%
131	25-40%	132	2.0-4.0%
219	25-40%	220	2.5-5.0%
414	1-3%		

4.4.3 In addition to the tabulated abundances listed above, other criteria should be reviewed at this time.

1) Ion peak widths: The appropriate range is between 0.5 and 0.6 AMU. AMU widths beyond this range can lead to loss of minor isotopes, while insufficient peak widths will result in decreased sensitivity.

2) Leak check: The abundances of ions 18, 28, and 32 (water, nitrogen and oxygen, respectively) should be less than 5 percent relative to 69. A level greater than 5 percent is indicative of a leak in the system.

4.4.4 Following the system tuning using FC-43, the GC/MS is then calibrated with BFB. A 1 or 2 µL aliquot of BFB is injected directly into the GC yielding a mass of 25 ng or 50 ng.

4.4.5 The ion abundances and ratios are checked against the criteria detailed below **or** alternatively, other criteria may be used (e.g., CLP, 524.2). The only exceptions are Army Corps and AFCEE, which specify that the table listed in SW 846 must be used unless project specific requirements allow for alternative criteria. If BFB meets the tuning criteria, then the system is determined to be calibrated. If BFB does not meet the tuning criteria, the GC/MS is re-tuned with FC-43 as described above.

4.4.6 SW 846 allows for the use of 5 - 50 ng of BFB. In the absence of specific recommendations on how to acquire the mass spectrum of BFB from the instrument manufacturer, SW 846 states that the following approach has been shown to be useful: the mass spectrum of BFB may be acquired in the following manner. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak. Alternatively, a single point scan of the apex may be used or other documented approaches suggested by the instrument manufacturer.

4.4.7 CLP requires that the three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan no more than 20 scans prior to the elution of BFB. Do not background subtract part of the BFB peak.

SW 846 Tuning Criteria

Mass	Abundance %
50	15 - 40% of mass 95
75	30 - 60% of mass 95
95	base peak, 100% relative abundance
96	5 - 9% of mass 95
173	< 2% of mass 174
174	> 50% of mass 95
175	5 - 9% of mass 174
176	>95%, < 101% of mass 174
177	5 - 9% of mass 176

CLP Tuning Criteria

Mass	Abundance %
50	8 - 40% of mass 95
75	30 - 66% of mass 95
95	base peak, 100% relative abundance
96	5 - 9% of mass 95
173	< 2% of mass 174
174	50-120% of mass 95
175	4 - 9% of mass 174
176	93 - 101% of mass 174
177	5 - 9% of mass 176

4.5 Initial Multi-Point Calibration

4.5.1 The first option is to begin with the simplest approach, the linear model through the origin using RSDs, as described in section 7.5 of 8000B. In methods where this is not appropriate because the calibration acceptance criteria of 15% for non-CCC compounds cannot be met, the next option of calibration (linear regression) is used. The other calibration option is use of the quadratic curve. This calibration option requires a minimum of 6 points and is used in cases where the detector doesn't meet calibration criteria over a wide range. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. (Designating sequences, the successive members of which either consistently increase or decrease but do not oscillate in relative value. Each member of a monotone increasing

sequence is greater than or equal to the preceding member; each member of a monotone decreasing sequence is less than or equal to the preceding member.)

4.5.2 The requirement for the linear or quadratic regression model is that R^2 be greater than or equal to 0.990. The difficulty with regression models is that the intercept may be greater than the detection limit. These calibration options exhibit a greater accuracy in the mid-range, and may demonstrate more extreme responses at the low and high ends of the calibration, and therefore, it is important to dilute samples and extracts so that they are in the mid-range of the calibration.

4.5.3 For linear calibrations using the RSD, analyze standard solutions using a minimum of 5 different concentration levels. The lowest concentration establishes the reporting limit, and the highest concentration defines the upper usable working range of the detector. However, there are some cases where the reporting limit may be higher than the lowest standard due to poor purging efficiency of the analyte. Criteria for evaluating these standards are detailed in Section 6.

4.5.4 After completion of a compliant initial calibration curve, an initial calibration verification (ICV) using standards from a different supplier is analyzed. The ICV must meet the client specific limits listed in Appendix XIV, but variations in standard sources may make it difficult to meet some client criteria. These analytes that do not meet client-specific criteria will be monitored for possible further action such as finding a third standard source. In some cases the analytes just have poor purging efficiencies. This second source independent calibration verification may be used as a CCV as long as it meets the limits specified in Section 6.

4.6 Continuing Calibration Verification

4.6.1 A CCV is analyzed once the BFB tuning criteria have been met and the instrument has been calibrated. Criteria for evaluating a CCV standard are detailed in Section 6.

4.7 Instrument Blank

4.7.1 The analysis of an instrument blank (IBLK) is performed prior to the injection of BFB. This analysis ensures that there are no volatile organic contaminants in either the purge and trap sample concentrator or in the autosampler.

4.7.2 Any sample that demonstrates concentrations of target analytes high enough to saturate the detector must be followed by at least one IBLK, also known as a saturation blank. This IBLK analysis is performed to prevent any possible carry-over into the subsequent

sample analysis. Evaluation criteria follow the same guidelines set forth for the analysis of a method blank.

4.8 Method Blank

4.8.1 After the analysis of the CCV and prior to sample analysis, a method blank must be analyzed. The analysis of the method blank demonstrates that the instrument is free of volatile organic contaminants and ensures that the reagent water is also free of contaminants. Refer to Section 6 for method blank acceptance criteria.

4.9 Sample Analysis

4.9.1 Samples (and associated QC) may be analyzed for up to 12 hours from the injection of BFB. The samples are analyzed subsequent to the calibration standard and method blank analyses. However, no more than twenty sample analyses are performed in a 12-hour period. Once all criteria for BFB, the calibration standard, and method blank have been met, the sample analyses are performed. The Eclipse sample concentrator purges both aqueous and soil samples at 40°C. The 4560 sample concentrator does not utilize a heated purge for aqueous samples.

4.10 Analysis sequence

IBLK (optional)
BFB
CCV/LCS
Method blank
Samples and QC

4.11 Sample Preparation

4.11.1 The samples are removed from the VOA refrigerator and are allowed to warm to room temperature. Aqueous samples, which are received in 40-mL vials, are placed onto the autosampler carousel or tray. Low-level soil samples (see section 1.4.2 of this SOP for further details on soil sample preservation) have 5 grams of sand added to them and are then placed onto the autosampler carousel or tray. See section 4.11.5 for the preparation of medium soils. The autosampler transfers a 5-mL aliquot of sample (or an aliquot of PFW for the soils) into the autosampler syringe, where it injects 1 µL of the internal standard and surrogate solution.

4.11.2 The pH of all water and preserved low level soil samples is measured subsequent to analysis in order to determine if they were preserved adequately without disturbing the

integrity of the sample. The pH values when not preserved to a pH ≤2 are noted in the instrument logbook and the client report. The project manager will also be notified immediately upon discovery of the out of control pH value.

4.11.3 If the concentration of any target analyte exceeds the initial calibration range, the sample must be diluted and reanalyzed.

4.11.4 If aqueous samples require dilutions, they are diluted by one of two methods listed:

1) the samples are diluted with PFW in a volumetric where they are transferred to 40 mL vials and placed onto the autosampler, or

2) the undiluted samples are placed on the autosampler, where they are diluted with PFW by the autosampler. Refer to the instrumentation manuals for the complete operation of the autosampler.

4.11.5 Medium Soils

4.11.6 The soil sample is gently mixed (no supernatant liquid is removed) and a 5 gram aliquot (weighed out to the nearest 0.1 g) is transferred to a tared 15 mL vial. 10.0 mL of methanol is added to the vial. The vial is sealed, then shaken for two minutes. A 100 µL sample aliquot per 5 mL of PFW is placed onto the autosampler carousel or tray. The autosampler transfers a volume of 5 mL of this sample into the autosampler syringe, where it injects 1 µL of the internal standard/ surrogate solution.

4.11.7 Soil samples with a significant moisture content (>10), that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of solvent/ water mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture determination. Laucks uses this calculation for all medium-level soil analyses.

4.12 Qualitative Identification

4.12.1 Two criteria must be satisfied to verify the identification of target compounds:

1) elution of the sample component within 0.50 minutes of the standard component and,

2) appropriate correspondence between the sample mass spectrum and the standard mass spectrum.

a) The three ions of greatest relative intensity, or any ions over 30% relative intensity that are present in the standard mass spectrum must be present in the sample spectrum.

b) The relative intensities of ions must be within 30% between the standard and sample spectra.

d) The emphasis of spectral review is on the three major ions. However, if a target compound cannot be verified by all the criteria listed above, but, in the technical judgment of the analyst, the identification is correct, then the analyte will be quantitated, and reported. Compounds meeting the identification criteria must be reported with their spectra.

4.12.2 A library search may be performed for the purpose of identifying up to 10 largest non-target analytes also referred to as Tentatively Identified Compounds (TICs) if requested by the client. The criteria for confirming TICs are listed below.

1) Relative intensities of ions in the reference spectrum > 10% of the most abundant ion should be present in the sample spectrum.

2) Molecular ions in the reference spectrum should be present in the sample spectrum.

3) The relative intensities for the major ions should agree within 20%.

4) Ions in the sample spectrum but not present in the standard spectrum should be reviewed for possible background contamination or coelution.

6) If, after careful review and in the technical judgment of the analyst, no valid tentative identification can be made, the compound should be reported as follows:

A) If the library search produces a match at or above 85%, report that compound.
B) If the library search produces more than one compound at or above 85%, report the first compound.
C)If the library search produces no matches at or above 85%, report the compound as unknown. The analyst should give additional classification of the unknown compound, if possible (i.e., unknown hydrocarbon, unknown acid type, unknown chlorinated compound).

4.13 Manual Peak Integrations

4.13.1 Manual peak integrations should be used only when necessary to correct for matrix interference, tangent peaks, split peaks, overloaded peaks and rising baselines. Manual

integrations are not to be used in an attempt to meet calibration, surrogate recovery, or spike recovery criteria. Refer to LTL-1020, *The Integration of IC, GC, HPLC, and GC/MS Peaks* for more detail.

4.13.2 If a manual integration is necessary, follow the following guidelines:

1) Integrate only the peak. Start where the peak lifts from the baseline and end as soon as it returns to the baseline. Do not integrate extra baseline in an attempt to increase peak area.

2) Do not "peak shave". Do not cut off legitimate parts of the peak in order to reduce peak area.

3) In cases of tangent skims, do not increase or decrease peak areas or heights by skimming extra long baselines or drawing the baseline too low.

4) Always initial and date your manual integrations.

4.14 Compound Quantification

4.14.1 Target compound concentrations are calculated using the following equations:

Aqueous samples:

$$\text{Conc } (\mu g / L) = \frac{A_x \times I_s \times Df}{A_{is} \times ARF \times V_o}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average RF from the ambient temperature purge of the initial calibration.
V_o = Volume of water purged (mL).
Df = Dilution factor. Assume 1.0 for no dilution.

Low soil (on a dry-weight basis)

$$\text{Conc } (\mu g / kg) = \frac{A_x \times I_s}{A_{is} \times ARF \times W_s \times D}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average response factor from the heated purge of the initial calibration.
W_s = Weight of sample added to purge vessel (g).

$$D = \frac{100 - \%\text{Moisture}}{100}$$

Medium soil (on a dry weight basis)

$$\text{Conc } (\mu g / kg) = \frac{A_x \times I_s \times V_t \times 1000 \times Df}{A_{is} \times ARF \times V_a \times W_s \times D}$$

where:

A_x = Area of the characteristic ion (EICP) of the analyte.
A_{is} = Area of the characteristic ion (EICP) of the internal standard.
I_s = Amount of the internal standard (ng).
ARF = Average RF from the ambient temperature purge of the calibration standard.
V_t = Total volume of the methanol extract in milliliters (10 mL+soil moist. correction vol.).
V_a = Volume of the methanol extract in microliters added to the reagent water for purging.
W_s = Weight of sample added to purge vessel (g).
Df = Dilution factor. Assume 1.0 for no dilution.

Medium soil moisture correction

mL (to be added to methanol volume) = (sample wt.) x (%moisture/100%)

5. Reports

5.1 Data Packet Organization

5.1.1 See Appendix II for a checklist detailing data packet organization.

5.2 Quality Control Reports

5.2.1 All results for quality control tests are entered into the lab database. Printouts of all data entered must be included in the data packet. The routine minimum is a method blank report, a blank spike report, and an MS/MSD report.

5.3 Control Limits

5.3.1 The laboratory has a computerized database that is used to generate control limits for various analyses, matrices, and analytes. The control limits define the range in which 99% of all values produced by a system should lie. Because the control limits are updated periodically, the current control limits listed in this SOP (Appendix XIII) may change. A copy of all current control limits is located in Laucks' Control Limits Database.

5.4 Data Qualifying Flags

5.4.1 Sample report results are qualified with data qualifying flags. These flags have the following definitions:

Code	Definition
U	The analyte of interest was not detected, to the limit of detection indicated.
B	The analyte of interest was detected in the method blank associated with the sample, as well as in the sample itself. The B flag is applied without regard to the relative concentrations detected in the blank and sample.
J	The analyte of interest was detected below the practical quantitation limit. This value should be regarded as an estimate.
D	The value reported is derived from the analysis of a diluted sample or sample extract.
E	The value reported is based on a sample or sample extract in which the target analyte concentration exceeded the calibration range. The value reported

Code	Definition
	should be considered an estimate.
N	Used to denote the presumptive evidence of a target analyte. This flag is used when it is not possible to confirm the presence of a compound by the strict guidelines for mass spectral interpretation, but in the technical judgment of the analyst, this compound is present.

*Alternate flags may be used, which are fully defined in the associated agency QAP or QSM.

6. Quality Control

6.1 Initial Calibration

6.1.1 Criteria

6.1.1.1 Initial calibration data are evaluated using %RSD and the relative response factors.

RFs are calculated using the equation:

$$RF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

where:

A_x = Response of target analyte
A_{is} = Response of internal standard
C_x = Amount of target analyte (ng/mL).
C_{is} = Amount of internal standard (ng/mL).

%RSDs are calculated using the equation:

$$\%RSD = \frac{SD_i}{RF_i} \times 100$$

where:

Sd_i = Standard deviation of the initial calibration points
RF_i = Average RF from initial calibration

6.1.2 The System Performance Check Compounds (SPCCs) are checked to determine that the minimum average relative response factors tabulated below are met, and that the maximum RSD for each Calibration Check Compound (CCC) is ≤ 30%. The RSD for all other target analytes *should* be ≤ 15%. If the RSD of any target analyte is greater than 15%, the following steps are taken: 1) Check instrument parameters and operating conditions. 2) Check to make sure that the problem is not associated with one of the six standards in which case that standard could be replaced. 3) Narrow the calibration range by dropping the lowest(changes the reporting limit) or highest calibration standard. A mid-ranged standard can not be dropped. After these steps have been taken and an analyte still exceeds 15% additional calibration options mentioned in section 4.5 may be used. Alternatively, if the mean of the RSD values for all analytes in the calibration is ≤ 15% with each individual analyte ≤ 30%, the initial calibration is considered acceptable. The Army Corps feels that by using the steps mentioned above, all of their target compounds should be less than 15%.

6.1.2.1 The mean RSD criterion applies to all analytes in the standards, regardless of whether or not they are of interest for a specific project.

6.1.2.2 The data used must be supplied with either a summary of the initial calibration data or a specific list of those compounds for which the RSD exceeded 15% and the results of the mean RSD calculation.

SPCC Compounds	Minimum RF Limit
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
Chlorobenzene	0.300
1,1,2,2-Tetrachloroethane	0.300

CCC Compounds	%RSD Limit
Vinyl chloride	±30%
1,1-Dichloroethene	±30%
Chloroform	±30%
1,2-Dichloropropane	±30%
Toluene	±30%
Ethylbenzene	±30%

6.1.3 Corrective action

6.1.3.1 If the criteria are not met, additional standards may be analyzed or appropriate instrument maintenance and analysis of new standards should be performed.

6.1.4 Documentation

6.1.4.1 Copies of the initial calibration standards and the calculated RRFs and RSDs are stored with the raw data. If criteria are not met, reanalysis of additional standard(s) are noted in the instrument run log.

6.2 Continuing Calibration Verification

6.2.1 Criteria

6.2.1.1 At the beginning of each 12-hour QC period, a CCV standard is analyzed. The RRF for each compound is calculated and the percent difference is calculated as follows:

$$\%D = \frac{RF_i - RF_c}{RF_i} \times 100$$

where:

RF_i = Average RF from Initial Calibration
RF_c = RF from CCV standard
%D = Percent difference

For analytes with a linear regression or quadratic regression curve fit the %Drift is calculated as follows:

$$\%\text{Drift} = \frac{\text{Calculated concentration} - \text{Theoretical concentration}}{\text{Theoretical concentration}} \times 100$$

6.2.1.2 The minimum RRFs for the SPCCs must meet the criteria applied in the initial calibration detailed above. The %D for the CCCs cannot exceed the 20% CCV criteria as tabulated below.

*CCC Compounds	% D Limit
1,1-Dichloroethene	±20%
Chloroform	±20%
1,2-Dichloropropane	±20%
Toluene	±20%
Ethylbenzene	±20%
Vinyl chloride	±20%

6.2.1.3 *According to the US ACOE Shell, Section I.9.2.5.4: "Evaluate the concentration of each target analyte and surrogate in the CCV. Verify that the percent drift or percent difference for the CCCs and all project-specified contaminants of concern are within ± 20 percent of their expected values. Evaluate remaining target analytes to assess instrument stability and survey the need for performing instrument maintenance." It is recommended that the list of project-specified contaminants of concern be made available to the laboratory.

6.2.1.4 The internal standard area responses must be within a factor of two (-50% to +100%) from that of the mid-point standard level of the most recent initial calibration. In addition, the retention time must fall within 30 seconds from that in the mid-point standard level of the most recent calibration sequence.

6.2.1.5 In some cases a subset of analytes (project-specified contaminants of concern) may have further requirements. If such limits are indicated, the client must provide a list of these requirements before the project begins.

6.2.2 Corrective action

6.2.2.1 Check calculations, check standard solutions or perform instrument maintenance. Reanalyze the standard, if this fails, re-do initial calibration.

6.2.3 Documentation

6.2.3.1 Copies of the continuing calibration standards and the calculated RFs, %Ds and %drifts are stored with the raw data. If criteria are not met, reanalysis of additional standard(s) are noted in the instrument run log.

6.3 Instrument Blank

6.3.1 Criteria

6.3.1.1 The criteria used are the same as those used for the method blank control limits.

6.3.2 Corrective action

6.3.2.1 If the initial IBLK contains measurable levels of target analytes above one-half the reporting limit, except where noted below, the system is out of control. The source of contamination must be identified and corrected. If analysis of an additional IBLK does not demonstrate that the analytical system is free of contaminants, then instrument maintenance such as cleaning the transfer lines or replacing the trap may be required.

6.3.3 Documentation

6.3.3.1 Copies of instrument blank analyses are stored with the sample analyses raw data. Re-analysis of the method blank and/or maintenance performed is documented in the instrument run log.

6.4 Method Blanks

6.4.1 Criteria

6.4.1.1 A method blank is an aliquot of PFW (with 5g of sand for low-level soils). It is used to verify contamination free reagents and apparatus. A method blank is analyzed after every CCV and prior to sample analysis. The autosampler draws 5mL from the blank water reservoir and directly injects internal standards and surrogates. Method blank criteria are listed below.

6.4.1.2 Laucks criterion for a method blank is less than one-half the RL for each analyte.

6.4.2 Corrective action

6.4.2.1 When these criteria are exceeded, corrective action should be taken to find/reduce/eliminate the source of this contamination in the method blank. Sample corrective action may be limited to qualification for blank contamination. When the concentrations of any target analytes within the method blank are above one-half of the reporting limit, the effect this may have had on the samples will be assessed as detailed below:

1) If any analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps shall be taken to find/reduce/eliminate the source of this contamination in the method blank. The case narrative should also discuss the situation.

2) If an analyte is found in the method blank and some, or all, of the other batch samples, additional corrective action is required to reanalyze the method blank, and any samples containing the same contaminant(s) which exceeded the corrective action limit(s). If the contamination remains, the contaminated samples of the batch would be reprepared and reanalyzed with a new method blank and batch specific QC samples.

3) Sporadic cases of contamination may be difficult to control however, daily contamination would not be acceptable.

4) Out-of-control surrogate recoveries in the method blank require reanalysis of the method blank.

5) If common lab contaminants such as acetone and methylene chloride are present, the associated sample results for these compounds are flagged appropriately. Bromomethane, 2-butanone, and methyl acetate may also be present in a medium soil method blank. Bromomethane is due to the build-up of methanol on the trap and is very difficult to control. 2-Butanone and methyl acetate are common methanol contaminants and are often present at very low levels. Analytes may be present as long as they are less than 5 percent of the regulatory limit associated with that analyte, or less than 5 percent of the sample result for the same analyte, whichever is greater, for the method blank to be acceptable. Typically, the regulatory limits for these compounds are well above normal sporadic contamination levels however, in order to make appropriate determinations in accordance with the DoD QSM and SW 846, the lab must have the specific regulatory limit for the common lab contaminants provided by the contractor or oversight agency.

6.4.3 Documentation

6.4.3.1 Copies of all method blank analyses are stored with the sample analyses raw data. In addition, the raw data for the method blank analyses are stored in the laboratory for an extended period of time. The results for the method blank analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank(s) are noted in the instrument run log. Outliers are discussed in the case narrative.

6.5 Blank Spikes

6.5.1 Criteria

6.5.1.1 A blank spike follows the same protocol as a matrix spike analysis except that the spiking solution is added to an aliquot of PFW (with sand for soil samples) instead of an actual sample. A method blank with added analytes is a blank spike. A blank spike is the same as a QC check standard. See Appendix XIII for laboratory control limits on the spike recoveries.

6.5.1.2 The USACOE requires at a minimum that all target analytes be spiked into the LCS. According to the USACOE Shell Document I.10.2.2: "The LCS is spiked with all single-component target analytes before it is carried through the preparation, cleanup, and determinative procedures. ***A subset of the single-component target analytes containing the specific analytes of interest can be substituted for the full list of target analytes if specified in project-specific contracts or work plans.***"

6.5.1.3 It is also recommended that all target analytes of concern be spiked into the LCS for non-USACE projects. NELAP specifies in D.1.1.b.4.: "if the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method." The DOD QSM Final Version III specifies, in clarification Box D-6: "all target analytes are spiked in the LCS. Target analytes are defined by the project or in Appendix DoD-C."

6.5.1.4 The level spiked in the blank spike will periodically be varied within the established calibration range.

6.5.2 Corrective action

6.5.2.1 The blank spike is used to determine whether a method is in control during sample preparation and analysis. An out of control blank spike would trigger sample reanalysis only if the sample surrogate recoveries and MS/MSD spike recoveries indicated sample processing errors.

6.5.3 Documentation

6.5.3.1 The raw data for the blank spike analyses are stored in the laboratory for an extended period of time. The results for the blank spike analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis of additional blank spike(s) are noted in the instrument run log.

6.6 Matrix Spike

6.6.1 Criteria

6.6.1.1 A sample is chosen at random (unless specified by a client) from the samples to be analyzed, and an aliquot of spiking solution is added to this sample prior to analysis. A matrix spike analysis is performed with each analytical batch, up to a maximum of 20 samples, unless there is insufficient sample provided by the client (Laucks informs all clients that extra sample volume must be submitted for MS/MSD analyses). The matrix spike sample is used to evaluate the matrix effect of the sample upon recovery of the analytes. The recovery of spike analytes is calculated as follows:

$$Recovery = \frac{SS - S}{SpikedAmount} \times 100$$

where:

SS = concentration in spiked sample
S = native concentration in unspiked sample

6.6.2 Corrective action

6.6.2.1 Samples with spike recoveries outside control limits (see Appendix XIII) will be reviewed for possible corrective action. Corrective action may involve recalculation and/or reanalysis. This process should also include evaluation of the recovery of surrogate compounds in the MS sample and recovery of matrix spiking compounds from the blank spike analysis. In all cases a narrative explanation of the condition is required to detail the corrective actions taken.

6.6.3 Documentation

6.6.3.1 The raw data for the matrix spike analyses are stored with the sample analyses raw data. The results for the matrix spike analyses are reported electronically via the laboratory's LIMS database.

6.7 Matrix Spike Duplicate

6.7.1 Criteria

6.7.1.1 A matrix spike duplicate analysis is performed with each analytical batch, up to a maximum of 20 samples. The compound recovery criteria are identical to those for the matrix spike sample. In addition, the matrix spike duplicate is used to measure method precision. This is done by computing the relative percent difference (RPD) between the matrix spike and matrix spike duplicate recovery values. This calculation is as follows:

$$RPD = \frac{S1 - S2}{(S1 + S2) \backslash 2} \times 100$$

where:

S1 = measured concentration for MS sample
S2 = measured concentration for MSD sample

6.7.2 Corrective action

6.7.2.1 If a trend in out-of-control RPD values is observed, the method used must be examined to determine the source of variance. Once this source is identified, the method must be changed so that samples can be analyzed with a predictable reproducibility.

6.7.3 Documentation

6.7.3.1 The raw data for the matrix spike duplicate analyses are stored with the sample analyses raw data. The results for the matrix spike duplicate analyses are reported electronically via the laboratory's LIMS database. Any out-of-control results are documented in the LIMS database and/or analytical narrative.

6.8 Surrogate Recovery

6.8.1 Criteria

6.8.1.1 Surrogates are chemically similar compounds added to every sample, method blank, and QC sample prior to sample processing. They are used to monitor potential sample processing errors and matrix effects. Appendix XIII lists all surrogates and their recovery limits. Surrogate compound recoveries are calculated as follow:

$$\text{Recovery} = \frac{S_m}{S_a} \times 100$$

where:

S_m = concentration of surrogate measured in sample
S_a = concentration of surrogate added

6.8.2 Corrective Action

6.8.2.1 Check calculations for possible error. Reanalysis is required for all surrogate recoveries that exceed the established control limits.

6.8.2.2 Out-of-control surrogate recoveries in the method blank require that the method blank be reanalyzed. Sample analyses are not performed until all criteria are met in the method blank analysis. Sample and QC analyses require reanalysis for out-of-control surrogate recoveries. If the sample reanalyses also demonstrate out-of-control surrogate recoveries due to matrix interference, this corrective action is discussed in the narrative.

6.8.3 Documentation

6.8.3.1 The surrogate recoveries for all samples, blanks, and QC analyses are reported electronically via the laboratory's LIMS database. If criteria are not met, reanalysis for out-of-control surrogate recoveries are designated by an "RE" in the instrument run log.

7. References

1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 8260B, 3rd edition, 2nd revision, December 1996.

2) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 8000C, 3rd revision, March 2003.

3) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5000B, 3rd edition, revision 0, December 1996.

4) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5030B, 3rd edition, 2nd revision, December 1996.

5) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), US EPA Office of Solid Waste and Emergency Response, - Method 5035A, Draft Revision 1, July 2002.

6) Hewitt, Alan "Frozen Storage of Soil Samples for VOC Analysis," Environmental Testing and Analysis, Sept/Oct 1999, Vol. 8, No.5.

7) Sorini, Susan, et al, "Evaluation of VOC Loss from Soil Samples:," Contaminated Soil Sediment and Water, April/May 2002.

8) National Environmental Laboratory Accreditation Program, July 1, 1999.

9) Department Of Defense Quality Systems Manual For Environmental Laboratories, Final Version 3, March 2005.

10) Air Force Center For Environmental Excellence, Quality Assurance Project Plan, Final Version 4.0.01, May 2005.

11) US ACE EM 200-1-3, Shell For Analytical Chemistry Requirements, February 2001.

APPENDIX I

Initial Calibration Concentrations For Waters (µg/L) And Medium Soils (µg/Kg)

Routine Analytes

Compound	STD1	STD2	STD3	STD4	STD5	STD6
Dichlorodifluoromethane	1	5	10	50	100	200
Chloromethane	1	5	10	50	100	200
Vinyl Chloride	1	5	10	50	100	200
Bromomethane	1	5	10	50	100	200
Chloroethane	1	5	10	50	100	200
Trichlorofluoromethane	1	5	10	50	100	200
1,1-Dichloroethene	1	5	10	50	100	200
Acetone	-	5	20	50	100	200
Carbon disulfide	1	5	10	50	100	200
Methylene chloride	1	5	10	50	100	200
trans-1,2-Dichloroethene	1	5	10	50	100	200
1,1-Dichloroethane	1	5	10	50	100	200
Vinyl acetate	1	5	10	50	100	200
2,2-Dichloropropane	1	5	10	50	100	200
cis-1,2-Dichloroethene	1	5	10	50	100	200
2-Butanone	-	5	10	50	100	200
Bromochloromethane	1	5	10	50	100	200
Chloroform	1	5	10	50	100	200
1,1,1-Trichloroethane	1	5	10	50	100	200
Carbon tetrachloride	1	5	10	50	100	200
1,1-Dichloropropene	1	5	10	50	100	200
Benzene	1	5	10	50	100	200
1,2-Dichloroethane	1	5	10	50	100	200
Trichloroethene	1	5	10	50	100	200
1,2-Dichloropropane	1	5	10	50	100	200
Dibromomethane	1	5	10	50	100	200
Bromodichloromethane	1	5	10	50	100	200
cis-1,3-Dichloropropene	1	5	10	50	100	200
4-Methyl-2-pentanone	-	5	10	50	100	200
Toluene	1	5	10	50	100	200
trans-1,3-Dichloropropene	1	5	10	50	100	200
1,1,2-Trichloroethane	1	5	10	50	100	200
Tetrachloroethene	1	5	10	50	100	200
1,3-Dichloropropane	1	5	10	50	100	200

Compound	STD1	STD2	STD3	STD4	STD5	STD6
2-Hexanone	-	5	10	50	100	200
Chlorodibromomethane	1	5	10	50	100	200
1,2-Dibromoethane	1	5	10	50	100	200
Chlorobenzene	1	5	10	50	100	200
1,1,1,2-Tetrachloroethane	1	5	10	50	100	200
Ethylbenzene	1	5	10	50	100	200
m,p-Xylene	2	10	20	100	200	400
o-Xylene	1	5	10	50	100	200
Styrene	1	5	10	50	100	200
Bromoform	1	5	10	50	100	200
Isopropylbenzene	1	5	10	50	100	200
Bromobenzene	1	5	10	50	100	200
1,2,3-Trichloropropane	1	5	10	50	100	200
1,1,2,2-Tetrachloroethane	1	5	10	50	100	200
n-Propylbenzene	1	5	10	50	100	200
2-Chlorotoluene	1	5	10	50	100	200
4-Chlorotoluene	1	5	10	50	100	200
1,3,5-Trimethylbenzene	1	5	10	50	100	200
tert-Butylbenzene	1	5	10	50	100	200
1,2,4-Trimethylbenzene	1	5	10	50	100	200
sec-Butylbenzene	1	5	10	50	100	200
1,3-Dichlorobenzene	1	5	10	50	100	200
1,4-Dichlorobenzene	1	5	10	50	100	200
p-Isopropyltoluene	1	5	10	50	100	200
1,2-Dichlorobenzene	1	5	10	50	100	200
n-Butylbenzene	1	5	10	50	100	200
1,2-Dibromo-3-chloropropane	1	5	10	50	100	200
1,2,4-Trichlorobenzene	1	5	10	50	100	200
Naphthalene	1	5	10	50	100	200
Hexachlorobutadiene	1	5	10	50	100	200
1,2,3-Trichlorobenzene	1	5	10	50	100	200
Methyl tert-butyl ether	1	5	10	50	100	200
1,1,2-Trichloro-1,2,2-trifluoromethane	1	5	10	50	100	200
Methyl acetate	-	5	10	50	100	200
Cyclohexane	1	5	10	50	100	200
Methylcyclohexane	1	5	10	50	100	200
1-Chlorohexane	1	5	10	50	100	200

Initial Calibration Concentrations Of Routine Target Analytes For Low Level Soils

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5	STD6
Dichlorodifluoromethane	3	10	20	50	100	200
Chloromethane	3	10	20	50	100	200
Vinyl Chloride	3	10	20	50	100	200
Bromomethane	3	10	20	50	100	200
Chloroethane	3	10	20	50	100	200
Trichlorofluoromethane	3	10	20	50	100	200
1,1-Dichloroethene	3	10	20	50	100	200
Acetone	-	10	20	50	100	200
Carbon disulfide	3	10	20	50	100	200
Methylene chloride	3	10	20	50	100	200
trans-1,2-Dichloroethene	3	10	20	50	100	200
1,1-Dichloroethane	3	10	20	50	100	200
cis-1,2-Dichloroethene	3	10	20	50	100	200
2-Butanone	-	10	20	50	100	200
Chloroform	3	10	20	50	100	200
1,1,1-Trichloroethane	3	10	20	50	100	200
Carbon tetrachloride	3	10	20	50	100	200
Benzene	3	10	20	50	100	200
1,2-Dichloroethane	3	10	20	50	100	200
Trichloroethene	3	10	20	50	100	200
1,2-Dichloropropane	3	10	20	50	100	200
Bromodichloromethane	3	10	20	50	100	200
cis-1,3-Dichloropropene	3	10	20	50	100	200
4-Methyl-2-pentanone	-	10	20	50	100	200
Toluene	3	10	20	50	100	200
trans-1,3-Dichloropropene	3	10	20	50	100	200
1,1,2-Trichloroethane	3	10	20	50	100	200
Tetrachloroethene	3	10	20	50	100	200
2-Hexanone	-	10	20	50	100	200
Chlorodibromomethane	3	10	20	50	100	200
Chlorobenzene	3	10	20	50	100	200
Ethylbenzene	3	10	20	50	100	200
m,p-Xylene	6	20	40	100	200	400
o-Xylene	3	10	20	50	100	200
Styrene	3	10	20	50	100	200

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5	STD6
Bromoform	3	10	20	50	100	200
1,1,2,2-Tetrachloroethane	3	10	20	50	100	200

Initial Calibration Concentrations Of Additional Analytes For Low Soils

Compound (µg/L)	STD1	STD2	STD3	STD4	STD5	STD6
1,1,1,2-Tetrachloroethane	3	10	20	50	100	200
2,2-Dichloropropane	3	10	20	50	100	200
Bromochloromethane	3	10	20	50	100	200
1,1-Dichloropropene	3	10	20	50	100	200
1,2-Dibromoethane	3	10	20	50	100	200
Dibromomethane	3	10	20	50	100	200
1,3-Dichloropropane	3	10	20	50	100	200
Isopropylbenzene	3	10	20	50	100	200
Bromobenzene	3	10	20	50	100	200
1,2,3-Trichloropropane	3	10	20	50	100	200
n-Propylbenzene	3	10	20	50	100	200
2-Chlorotoluene	3	10	20	50	100	200
4-Chlorotoluene	3	10	20	50	100	200
1,3,5-Trimethylbenzene	3	10	20	50	100	200
tert-Butylbenzene	3	10	20	50	100	200
1,2,4-Trimethylbenzene	3	10	20	50	100	200
sec-Butylbenzene	3	10	20	50	100	200
1,3-Dichlorobenzene	3	10	20	50	100	200
1,4-Dichlorobenzene	3	10	20	50	100	200
p-Isopropyltoluene	3	10	20	50	100	200
1,2-Dichlorobenzene	3	10	20	50	100	200
n-Butylbenzene	3	10	20	50	100	200
1,2-Dibromo-3-chloropropane	3	10	20	50	100	200
1,2,4-Trichlorobenzene	3	10	20	50	100	200
Naphthalene	3	10	20	50	100	200
Hexachlorobutadiene	3	10	20	50	100	200
1,2,3-Trichlorobenzene	3	10	20	50	100	200
Iodomethane	3	10	20	50	100	200
1,1,2-Trichloro-1,2,2-trifluoromethane	3	10	20	50	100	200
Methyl acetate	-	10	20	50	100	200
Cyclohexane	3	10	20	50	100	200
Methylcyclohexane	3	10	20	50	100	200
1-Chlorohexane	3	10	20	50	100	200

APPENDIX II

Data Packet Sequence

1. QC Summary
   - Surrogate recoveries
   - MS/MSD summary
   - Method blank summary
   - Instrument performance check (tuning)
   - Internal standard areas and RT summary

2. Sample Data
   - Target compound results
   - TICs (if requested)
   - Chromatogram normalized to non-solvent peak
   - Quantitation report
   - Spectra

3. Standards Data
   - Initial calibration form
   - Chromatograms
   - Quantitation reports
   - CCV form
   - Chromatograms
   - Quantitation reports

4. Raw QC
   - BFB tune spectrum, chromatogram, and tabular listing
   - Method blank results, TICs, chromatogram, quantitation report, and spectra
   - MS/MSD results, chromatogram, quantitation report, spectra and control charts

5. Bench Sheets
   - Copy of instrument logbook, copy of COC, copy of holding blank analysis

6. Narrative

7. Electronic Disk Deliverables (EDD) when requested

8. Control charts

APPENDIX III

Determination of Vinyl Chloride by Selected Ion Monitoring (SIM)

Analytical Conditions:*

GC Parameters

Injection Port Temperature	: 200°C
GC/MS Interface Temperature	: 200°C
Initial GC Temperature	: 35°C
Initial Hold Time	: 1 min
Ramp Rate	: 14°C/min
Final Temperature	: 160°C
Final Time	: 3 min
Carrier Gas Flow	: 15 mL/min
Column Head Pressure	: 9 psi

Purge and Trap Parameters

Purge Time	: 11 min
Desorb Time	: 1 min
Bake Time	: 6 min
Desorb Preheat Temperature	: 20°C
Desorb Temperature	: 225°C
Purge Flow	: 40 mL/min
Purge Temperature	: Ambient
Desorb Flow Rate	: 15 mL/min

Mass Spectrometer Parameters

Electron Energy	: 70 volts
Acquisition Start Time	: 0.1 min

*parameters may vary per instrument

Group 1:

M/Z	Dwell Time (milliseconds)
61	200
62	400
63	200
64	200

Group 2:

M/Z	Dwell Time (milliseconds)
49	200
51	200
128	100
129	200

Group 3:

M/Z	Dwell Time (milliseconds)
65	200
67	200
102	200
104	200

Standards:

Internal Standard: Bromochloromethane
Surrogate: 1,2-Dichloroethane-d4

APPENDIX IV

Appendix IX Compounds And Their Initial Concentrations

Waters

Compound* (µg/L)	STD1	STD2	STD3	STD4	STD5
Acrolein	5	10	50	100	200
Bromoethane	5	10	50	100	200
Allyl chloride	5	10	50	100	200
Acrylonitrile	5	10	50	100	200
Chloroprene	5	10	50	100	200
Methacrylonitrile	50	100	500	1000	2000
Ethyl methacrylate	5	10	50	100	200
trans-1,4-Dichloro-2-butene	5	10	50	100	200
2-Chloroethylvinyl ether[1]	5	10	50	100	200
Acetonitrile	50	100	500	1000	2000
Propionitrile	50	100	500	1000	2000
Isobutanol	100	200	1000	2000	4000

* The calibration range defines the reporting range for these compounds. However, the reporting limits may change for individual projects, based on MDLs, mass spectral confirmation, or project-specific requirements.

1 This analyte is not recommended because it decomposes in water, and does not recover in acidified samples.

Additional California Oxygenate Compounds And Their Initial Concentrations

Waters

Compound* (µg/L)	STD1	STD2	STD3	STD4	STD5	STD6
Tert-butanol	5	25	50	250	500	1000
Isopropyl ether*	1	5	10	50	100	200
Ethyl-tert butyl ether	1	5	10	50	100	200
Tert-amyl methyl ether	1	5	10	50	100	200

*co-eludes and interferes with vinyl acetate

APPENDIX V

Method 8260 Requirements and Corrective Actions

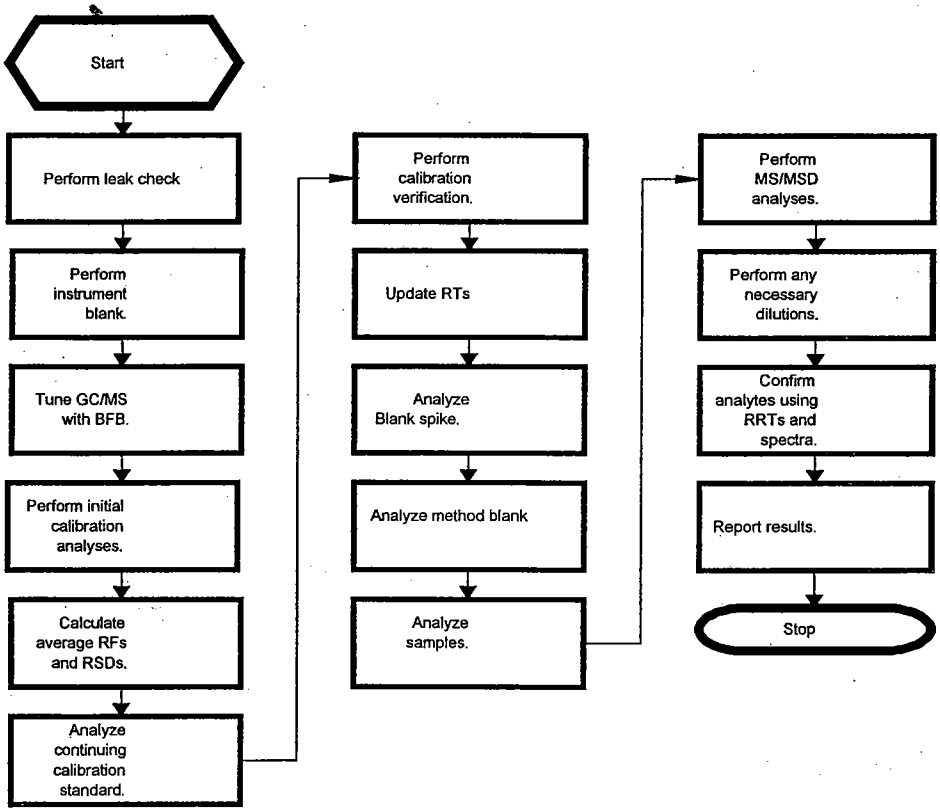
QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Holding Time	14 days from collection.	14 days from collection.	N/A	N/A	HTVR (holding time violation report).
Tuning Verification 5 - 200 ng BFB	Mass Abundance 50 15-40% of 95 75 30-60% of 95 95 100% 96 5-9% of 95 173 < 2% of 174 174 > 50% of 95 175 5-9% of 174 176 95-101% of 174 177 5-9% of 176 *Alternate tuning criteria may be used (e.g.,CLP,524.2).	Mass Abundance 50 15-40% of 95 75 30-60% of 95 95 100% 96 5-9% of 95 173 < 2% of 174 174 >50% of 95 175 5-9% of 174 176 95-101% of 174 177 5-9% of 176 * Alternate tuning criteria may be used (except Army Corps and AFCEE).	Every 12 hours.	Re-tune instrument to meet criteria.	Copy of BFB raw data and tune criteria with file.
Initial Calibration	Minimum of 5 levels with lowest near RL. %RSD for CCCs ≤30% RF for SPCCs: chloromethane 0.10 1,1-dichloroethane 0.10 bromoform 0.10 chlorobenzene 0.30 1,1,2,2-tetrachloroethane 0.30 Non-CCCs: all analytes or average of all analytes = ≤15%. Or use Linear or Quadratic Regression where $r^2 \geq 0.990$	Minimum of 5 levels %RSD for CCCs ≤30% RFs for SPCCs: chloromethane 0.10 1,1-dichloroethane 0.10 bromoform 0.10 chlorobenzene 0.30 1,1,2,2-tetrachloroethane 0.30 Non-CCCs: all analytes or average of all analytes ≤15% Or use Linear or Quadratic Regression where $r^2 \geq 0.990$	Initially.	Reanalysis of out of control standards.	Copies of all raw data, tune file, and Form VI.
Initial Calibration Verification		Typically mid-range standard. Client specific limits.	Once after initial calibration.	Re-analysis of initial calibration.	
Continuing Calibration Verification	Mid-level calibration standard every 12 hours. RF for SPCCs same as initial. RF for CCCs must be ≤20% D.	%D for CCCs ≤20% . RF for SPCCs same as initial.	Every 12 hours.	Reanalyze for analytes that exceeds the control limits.	Copies of raw data and Form VII.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Internal Standards	Fluorobenzene, chlorobenzene-d5, 1,4-dichlorobenzene-d4, recommended.	Fluorobenzene, chlorobenzene-d5 and 1,4-dichlorobenzene-d4. RT must be ± 30 seconds from last calibration; area must be 200 to 100%.	Every sample.	Reanalyze all samples that do not meet these criteria.	IS areas and RTs are submitted on Form 8, and included in data package.
Method Blank/ Instrument Blank	One method blank per extraction batch (up to 20 samples) or when there is a change in reagents, whichever is more frequent. The method blanks should b: less than the MDL, or less than level of acceptable blank contamination in QAPP, or <5% of the regulatory limit, or less than 5% of the sample result, whichever is greater.	< 1/2 Reporting limit.	One method blank per QC period, after the CCV, prior to sample analysis. Typically, an instrument blank is analyzed prior to tuning the instrument.	Evaluate, qualify.	Copies of raw data filed, and reported electronically. Daily control charts for all method blanks are recorded. Narrate any analytes >1/2 RL
Surrogate Standards	4-bromofluorobenzene, dibromofluoromethane 1,2-dichloroethane-d4 or toluene-d8 recommended. Compare %R to laboratory established limits.	1,2-dichloroethane-d4, toluene-d8, 4-bromofluorobenzene, and dibromofluoromethane. Percent recoveries must meet established laboratory control limits. See QC database for control limits.	Every sample.	Reanalysis if surrogate recoveries exceed the established control limits.	Surrogates that exceed the control limits are documented in associated file.
Quality Control	One MS/MSD per 20 samples or each batch of samples, whichever is more frequent. Compare % recovery to laboratory established limits.	Blank spike recoveries and RPDs must meet control limits established by laboratory. MS/MSD recoveries are allowed to be out if due to matrix effect. See QC database for control limits.	One MS/MSD per 20 samples. A blank spike is analyzed every QC period.	Reanalysis of MS/MSD unless matrix effect has already been demonstrated. This can be determined by comparing with the blank spike recoveries.	Blank spike and MS/MSD recoveries are stored electronically. Raw data is stored with file. Out of control events are noted in file.
Qualitative Identification	All ions > 10% intensity must be ± 20% of standard; ± 0.06 RRT units of standard RRT.	All ions > 10% intensity must be ± 20% of standard; ± 0.200 min. of standard RT.	For identifying all target analytes in each sample.		Copies of all mass spectra stored with data.

QA Element	Method Criterion	Laucks Criterion	Frequency	Corrective Action	Documentation
Sample Preservation and Storage	pH ≤2 with HCl for waters. pH ≤ 2 with sodium bisulfate for low level soils. Medium soils: zero headspace, methanol, or EnCore™ sampler. Store at 4 ° C.	pH ≤2 with HCl for waters. pH ≤2 with sodium bisulfate for low level soils. Medium soils: zero headspace, methanol, or EnCore™ sampler. Store at 4° C, Freezing for soils.	All water and low level soil samples.	If the water and low-level soil samples are not acid-preserved, this is narrated in final report and the project manager is notified.	pH of all water samples and low level soils, where not preserved to a pH of ≤2, noted in instrument logbook.
Standard Solution Expiration	Stock standards (except gases): 6 months. Store between -10 to -20° C in Teflon-sealed liners and amber bottles with minimal headspace, protect from light. Stock gas standards: 2 months. Storage conditions same as stock standards. Calibration standards: daily.	Stock standards (except gases): 6 months. Gas stock standards: 2 months. Stored at -10° to -20° C . Calibration standards: daily. All standards sealed in flame-sealed ampules: 2 years (not to exceed parent expiration date).			All standard data recorded in standard logbook.
Field Sample Amount	2 40 mL vials with zero headspace. Soils: vials, with zero headspace, or a sample aliquot in 40-mL vial. Glass container, septum-sealed screw caps.	Waters:4 40 mL vials with zero headspace. Soils: 2 oz vials, with zero headspace, or a sample aliquot in 4 -40 mL vial. Glass container, septum-sealed screw caps.			Bottles received by laboratory documented electronically.
Applicability	Groundwaters, soils, sediments, sludge, non-water miscible wastes and others.				

APPENDIX VI

Method 8260B Flow Chart



Refer to correctve action chart for out of control events.

APPENDIX VII

Method 8260 Target Analytes

Dichlorodifluoromethane	Chloromethane
Vinyl Chloride	Bromomethane
Chloroethane	Trichlorofluoromethane
1,1-Dichloroethene	Acetone
Carbon disulfide	Methylene chloride
trans-1,2-Dichloroethene	1,1-Dichloroethane
1,1,2,2-Tetrachloroethane	2,2-Dichloropropane
cis-1,2-Dichloroethene	2-Butanone
Bromochloromethane	Chloroform
1,1,1-Trichloroethane	Carbon tetrachloride
1,1-Dichloropropene	Benzene
1,2-Dichloroethane	Trichloroethene
1,2-Dichloropropane	Dibromomethane
Bromodichloromethane	cis-1,3-Dichloropropene
4-Methyl-2-pentanone	Toluene
trans-1,3-Dichloropropene	1,1,2-Trichloroethane
Tetrachloroethene	1,3-Dichloropropane
2-Hexanone	Chlorodibromomethane
1,2-Dibromoethane	Chlorobenzene
1,1,1,2-Tetrachloroethane	Ethylbenzene
m,p-Xylene	o-Xylene
Styrene	Bromoform
Methyl tert-butyl ether	1,1,2-Trichloro-1,2,2-trifluoromethane
Methyl acetate	Cyclohexane
Methylcyclohexane	

APPENDIX VIII

Method 8260 Optional Analytes for Low Level Water Analyses

Isopropylbenzene	Bromobenzene
1,2,3-Trichloropropane	2-Chlorotoluene
n-Propylbenzene	1,3,5-Trimethylbenzene
4-Chlorotoluene	1,2,4-Trimethylbenzene
tert-Butylbenzene	1,3-Dichlorobenzene
sec-Butylbenzene	p-Isopropyltoluene
1,4-Dichlorobenzene	n-Butylbenzene
1,2-Dichlorobenzene	1,2,4-Trichlorobenzene
1,2-Dibromo-3-chloropropane	Hexachlorobutadiene
Naphthalene	1,2,3-Trichlorobenzene
Vinyl acetate	Tert-butanol
Tert-amyl methyl ether	Isopropyl ether
Ethyl-tert butyl ether	1-Chlorohexane

Appendix IX

Additional Appendix IX Compounds

Acrolein	Bromoethane
Allyl chloride	Acrylonitrile
Chloroprene	Methacrylonitrile
Methyl methacrylate	trans-1,4-Dichloro-2-butene
2-Chloroethylvinyl ether	Ethyl methacrylate
Iodomethane	Acetonitrile
Propionitrile	Isobutanol

APPENDIX X

Target Analytes And Their Associated Internal Standards

Fluorobenzene	**Chlorobenzene-d5**	**1,4-Dichlorobenzene-d4**
Dichlorodifluoromethane	Toluene	Isopropylbenzene
Chloromethane	trans-1,3-Dichloropropene	Bromobenzene
Vinyl Chloride	1,1,2-Trichloroethane	1,2,3-Trichloropropane
Bromomethane	Tetrachloroethene	1,1,2,2-Tetrachloroethane
Chloroethane	1,3-Dichloropropane	n-Propylbenzene
Trichlorofluoromethane	2-Hexanone	2-Chlorotoluene
1,1-Dichloroethene	Chlorodibromomethane	4-Chlorotoluene
Acetone	1,2-Dibromoethane	1,3,5-Trimethylbenzene
Carbon disulfide	Chlorobenzene	tert-Butylbenzene
Methylene chloride	1,1,1,2-Tetrachloroethane	1,2,4-Trimethylbenzene
trans-1,2-Dichloroethene	Ethylbenzene	sec-Butylbenzene
1,1-Dichloroethane	m,p-Xylene	1,3-Dichlorobenzene
Vinyl acetate	o-Xylene	1,4-Dichlorobenzene
2,2-Dichloropropane	Styrene	p-Isopropyltoluene
cis-1,2-Dichloroethene	Bromoform	1,2-Dichlorobenzene
2-Butanone	1-Chlorohexane	n-Butylbenzene
Bromochloromethane		1,2-Dibromo-3-chloropropane
Chloroform		1,2,4-Trichlorobenzene
1,1,1-Trichloroethane		Naphthalene
Carbon tetrachloride		Hexachlorobutadiene
1,1-Dichloropropene		1,2,3-Trichlorobenzene
Benzene		
1,2-Dichloroethane		
Trichloroethene		
1,2-Dichloropropane		
Dibromomethane		
Bromodichloromethane		
cis-1,3-Dichloropropene		
4-Methyl-2-pentanone		
Methyl tert-butyl ether		
1,1,2-trichloro-1,2,2-trifluoromethane		
Methyl acetate		
Cyclohexane		
Methylcyclohexane		

Additional Appendix IX Compounds And Associated Internal Standard

Fluorobenzene (IS)	**Chlorobenzene-d5**
Acrolein	Ethyl methacrylate
Bromoethane	
Allyl chloride	
Acrylonitrile	
Chloroprene	
Methacrylonitrile	
Methyl methacrylate	
trans-1,4-Dichloro-2-butene	
Iodomethane	
2-Chloroethylvinyl ether	
Acetonitrile	
Propionitrile	
Isobutanol	

Additional California Oxygenate Compounds And Associated Internal Standard

Fluorobenzene (IS)
Tert-butanol
Isopropyl ether
Ethyl-tert butyl ether
Tert-amyl methyl ether

APPENDIX XI

Target Analytes And Their Quantitation Ions

Compound	Primary Quant Ions*	Secondary Quant Ions*
Dichlorodifluoromethane	85	87
Chloromethane	50	52
Vinyl Chloride	62	64
Bromomethane	96	94
Chloroethane	64	66
Trichlorofluoromethane	101	103
1,1-Dichloroethene	96	61,98
Acetone	43	58
Carbon disulfide	76	78
Methylene chloride	84	49,86
trans-1,2-Dichloroethene	96	98,61
1,1-Dichloroethane	63	65
2,2-Dichloropropane	77	79
cis-1,2-Dichloroethene	96	98,61
2-Butanone	43	57,72
Bromochloromethane	128	49,130
Chloroform	83	85
1,1,1-Trichloroethane	97	99
Carbon tetrachloride	117	119
1,1-Dichloropropene	75	77,110
Benzene	78	-
1,2-Dichloroethane	62	98
Trichloroethene	130	95,132
1,2-Dichloropropane	63	65
Dibromomethane	93	95,174
Bromodichloromethane	83	85
cis-1,3-Dichloropropene	75	77
4-Methyl-2-pentanone	43	58,85
Toluene	92	91
trans-1,3-Dichloropropene	75	77
1,1,2-Trichloroethane	97	99
Tetrachloroethene	166	164
1,3-Dichloropropane	76	78
2-Hexanone	43	58,100

Compound	Primary Quant Ions*	Secondary Quant Ions*
Chlorodibromomethane	129	127
1,2-Dibromoethane	107	109
Chlorobenzene	112	114
1,1,1,2-Tetrachloroethane	131	133
Ethylbenzene	91	106
m,p-Xylene	106	91
o-Xylene	106	91
Styrene	104	78
Bromoform	173	175
Isopropylbenzene	105	120
Bromobenzene	156	158
1,2,3-Trichloropropane	75	77
1,1,2,2-Tetrachloroethane	83	85
n-Propylbenzene	120	91
2-Chlorotoluene	91	126
4-Chlorotoluene	91	126
1,3,5-Trimethylbenzene	105	120
tert-Butylbenzene	119	91,134
1,2,4-Trimethylbenzene	105	120
sec-Butylbenzene	105	134
1,3-Dichlorobenzene	111	75,148
1,4-Dichlorobenzene	146	148,111
p-Isopropyltoluene	119	134,91
1,2-Dichlorobenzene	146	148,111
n-Butylbenzene	91	92
1,2-Dibromo-3-chloropropane	157	75,155
1,2,4-Trichlorobenzene	180	182
Naphthalene	128	127,129
Hexachlorobutadiene	225	223,227
1,2,3-Trichlorobenzene	180	182
Methyl tert-butyl ether	73	57
1,1,2-trichloro-1,2,2-trifluoromethane	101	85,151
Methyl acetate	43	74
Cyclohexane	56	69,84
Methylcyclohexane	83	55,98
1-Cholorohexane	91	55,41,69

Additional Appendix IX Compounds And Their Quantitation Ions

Compound	**Primary Quant Ions***	**Secondary Quant Ions***
Acrolein	56	55
Bromoethane	108	110
Allyl chloride	76	41,39,78
Acrylonitrile	53	52,51
Chloroprene	53	51,88,90
Methacrylonitrile	41	39,52,67
Ethyl methacrylate	69	41,86,99
Methyl methacrylate	41	39,69,100
trans-1,4-Dichloro-2-butene	53	75,88
Vinyl acetate	43	86
Iodomethane	142	127
Vinyl acetate	43	86
2-Chloroethylvinyl ether	63	65,106
Acetonitrile	40	41,39
Propionitrile	54	-
Isobutanol	43	41,31

* Primary and secondary quantitation ions assigned by Laucks may be different than those recommended in SW 846, in order to avoid interference.

Additional California Oxygenate Compounds And Their Quantitation Ions

Compound	Primary Quant Ions*	Secondary Quant Ions*
Tert-butanol	59	41
Isopropyl ether	45	87
Ethyl-tert butyl ether	59	87
Tert-amyl methyl ether	73	55,87

APPENDIX XII

Target Analytes, Their Elution Order And Standard Mixes

Compound	**Standard Mix**
Dichlorodifluoromethane	1
Chloromethane	1

Compound	Standard Mix
Vinyl Chloride	1
Bromomethane	1
Chloroethane	1
Trichlorofluoromethane	1
Acrolein	2
1,1-Dichloroethene	3
1,1,2-trichloro-1,2,2-trifluoromethane	3
Acetone	4
Iodomethane	4
Bromoethane	5
Carbon disulfide	4
Allyl Chloride	6
Acetonitrile	6
Methyl acetate	3
Methylene chloride	3
Methyl tert-butyl ether	8,9
Tert-butanol	8
trans-1,2-Dichloroethene	3
Acrylonitrile	2
1,1-Dichloroethane	3
Isopropyl ether	8
Vinyl acetate	4
Chloroprene	7
Ethyl-tert butyl ether	8
2,2-Dichloropropane	3
cis-1,2-Dichloroethene	3
Propionitrile	6
2-Butanone	4
Methacrylonitrile	6
Bromochloromethane	3
Chloroform	3
1,1,1-Trichloroethane	3
Cyclohexane	3
Carbon tetrachloride	3
Tert-amyl methyl ether	8
Methyl methacrylate	6
Isobutanol	6
1,1-Dichloropropene	3

Compound	Standard Mix
1,2-Dichloroethane-d4 (surr)	10
Benzene	3
1,2-Dichloroethane	3
Trichloroethene	3
Methylcyclohexane	3
1,2-Dichloropropane	3
Dibromomethane	3
Bromodichloromethane	3
2-Chloroethylvinyl ether	4
cis-1,3-Dichloropropene	3
Ethyl methacrylate	6
4-Methyl-2-pentanone	4
Toluene-d8 (surr)	11
Toluene	3
trans-1,3-Dichloropropene	3
1,1,2-Trichloroethane	3
Tetrachloroethene	3
1,3-Dichloropropane	3
2-Hexanone	4
Chlorodibromomethane	3
1,2-Dibromoethane	3
Chlorobenzene	3
1,1,1,2-Tetrachloroethane	3
Ethylbenzene	3
m,p-Xylene	3
o-Xylene	3
Styrene	3
Bromoform	3
Isopropylbenzene	3
4-Bromofluorobenzene (surr)	12
Bromobenzene	3
1,2,3-Trichloropropane	3
1,1,2,2-Tetrachloroethane	3
n-Propylbenzene	3
trans-1,4-Dichloro-2-butene	6
2-Chlorotoluene	3
4-Chlorotoluene	3
1,3,5-Trimethylbenzene	3

Compound	Standard Mix
tert-Butylbenzene	3
1,2,4-Trimethylbenzene	3
sec-Butylbenzene	3
1,3-Dichlorobenzene	3
1,4-Dichlorobenzene	3
p-Isopropyltoluene	3
1,2-Dichlorobenzene	3
n-Butylbenzene	3
1,2-Dibromo-3-chloropropane	3
1,2,4-Trichlorobenzene	3
Naphthalene	3
Hexachlorobutadiene	3
1,2,3-Trichlorobenzene	3
1-Chlorohexane	10

Key:

1 = 524.2 Volatile Gasses Crescent CC2006.10
2 = Acrolein/Acrylonitrile Crescent CC2098
3 = 8260 Volatiles Crescent CCS-743
4 = Appendix IX Ketones Crescent CC2239-10
5 = Ethyl bromide ChemService O-640 neat
6 = Appendix IX Vols Crescent CC2464.10
7 = Chloroprene Restek 30238
8 = Calif. Oxygenates Restek 30465
9 = MTBE Restek 30402
10 = Neat Material
11 = Neat Material
12 = Neat Material

At the time of this writing, these are the current standard solutions used for the calibration standards. However, these standards may change due to Vendor supply or project-specific requirements.

APPENDIX XIII

Reporting limits, MDLs, and Control Limits*

Water

Target Analyte	RL	MDL	Blank	MS/MSD			BS/BSD		
				LCL	UCL	RPD	LCL	UCL	RPD
Dibromochloromethane	1	0.21	0.5	60	135	30	60	135	
Dichlorodifluoromethane	1	0.75	0.5	30	155	30	30	155	
Chloromethane	1	0.24	0.5	40	125	30	40	125	
Vinyl chloride	1	0.27	0.5	50	145	30	50	145	
Bromomethane	1	0.52	0.5	30	145	30	30	145	
Chloroethane	1	0.3	0.5	60	135	30	60	135	
Trichlorofluoromethane	1	0.47	0.5	60	145	30	60	145	
1,1-Dichloroethene	1	0.3	0.5	70	130	30	70	130	
Acetone	5	0.52	2.5	40	140	30	40	140	
Carbon disulfide	1	0.33	0.5	35	160	30	35	160	
Methylene chloride	1	0.44	0.5	55	140	30	55	140	
trans-1,2-Dichloroethene	1	0.19	0.5	60	140	30	60	140	
1,1-Dichloroethane	1	0.16	0.5	70	135	30	70	135	
2,2-Dichloropropane	1	0.27	0.5	70	135	30	60	135	
cis-1,2-Dichloroethene	1	0.2	0.5	70	125	30	70	125	
2-Butanone	5	0.4	2.5	30	150	30	30	150	
Bromochloromethane	1	0.2	0.5	65	130	30	65	130	
Chloroform	1	0.23	0.5	65	135	30	65	135	
1,1,1-Trichloroethane	1	0.26	0.5	65	130	30	65	130	
Carbon tetrachloride	1	0.35	0.5	65	140	30	65	140	
1,1-Dichloropropene	1	0.26	0.5	75	130	30	75	130	
Benzene	1	0.17	0.5	80	120	30	80	120	
1,2-Dichloroethane	1	0.11	0.5	70	130	30	70	130	
Trichloroethene	1	0.18	0.5	70	125	30	70	125	
1,2-Dichloropropane	1	0.2	0.5	75	125	30	75	125	
Dibromomethane	1	0.15	0.5	75	125	30	75	125	
Bromodichloromethane	1	0.14	0.5	75	120	30	75	120	
cis-1,3-Dichloropropene	1	0.12	0.5	70	130	30	70	130	
4-Methyl-2-pentanone	5	0.094	2.5	60	135	30	60	135	
Toluene	1	0.15	0.5	75	120	30	75	120	
trans-1,3-	1	0.1	0.5	55	140	30	55	140	

Target Analyte	RL	MDL	Blank	MS/MSD			BS/BSD		
				LCL	UCL	RPD	LCL	UCL	RPD
Dichloropropene									
1,1,2-Trichloroethane	1	0.16	0.5	75	125	30	75	125	
Tetrachloroethene	1	0.24	0.5	45	150	30	45	150	
2-Hexanone	5	0.15	2.5	55	130	30	55	130	
1,3-Dichloropropane	1	0.1	0.5	75	125	30	75	125	
1,2-Dibromoethane	1	0.21	0.5	80	120	30	80	120	
Chlorobenzene	1	0.17	0.5	80	120	30	80	120	
Ethylbenzene	1	0.25	0.5	75	125	30	75	125	
1,1,1,2-Tetrachloroethane	1	0.18	0.5	80	130	30	80	130	
m,p-Xylene	2	0.48	0.5	75	130	30	75	130	
o-Xylene	1	0.32	0.5	80	120	30	80	120	
Styrene	1	0.19	0.5	65	135	30	65	135	
Bromoform	1	0.22	0.5	70	130	30	70	130	
Isopropylbenzene	1	0.37	0.5	75	125	30	75	125	
1,1,2,2-Tetrachloroethane	1	0.16	0.5	65	130	30	65	130	
n-Propylbenzene	1	0.26	0.5	70	130	30	70	130	
Bromobenzene	1	0.18	0.5	75	125	30	75	125	
1,2,3-Trichloropropane	1	0.3	0.5	75	125	30	75	125	
2-Chlorotoluene	1	0.31	0.5	75	125	30	75	125	
1,3,5-Trimethylbenzene	1	0.28	0.5	75	130	30	75	130	
4-Chlorotoluene	1	0.26	0.5	75	130	30	75	130	
tert-Butylbenzene	1	0.38	0.5	70	130	30	70	130	
1,2,4-Trimethylbenzene	1	0.32	0.5	75	130	30	75	130	
sec-Butylbenzene	1	0.33	0.5	70	125	30	70	125	
4-Isopropyltoluene	1	0.29	0.5	75	130	30	75	130	
1,3-Dichlorobenzene	1	0.28	0.5	75	125	30	75	125	
1,4-Dichlorobenzene	1	0.34	0.5	75	125	30	75	125	
n-Butylbenzene	1	0.29	0.5	70	135	30	70	135	
1,2-Dichlorobenzene	1	0.37	0.5	70	120	30	70	120	
1,2-Dibromo-3-chloropropane	1	0.22	0.5	50	130	30	50	130	
1,2,4-Trichlorobenzene	1	0.41	0.5	65	135	30	65	135	
Hexachlorobutadiene	1	0.55	0.5	50	140	30	50	140	
Naphthalene	1	0.43	0.5	55	140	30	55	140	
1,2,3-Trichlorobenzene	1	0.48	0.5	55	140	30	55	140	

Low level soil

Target Analyte	RL	MDL	Blank	MS/MSD			BS/BSD		
				LCL	UCL	RPD	LCL	UCL	RPD
Dibromochloromethane	3	0.25	1.5						
Dichlorodifluoromethane	3	1.1	1.5	35	135	30	35	135	
Chloromethane	3	0.8	1.5	50	130	30	50	130	
Vinyl chloride	3	0.77	1.5	60	125	30	60	125	
Bromomethane	3	0.72	1.5	30	160	30	30	160	
Chloroethane	3	0.67	1.5	40	155	30	40	155	
Trichlorofluoromethane	3	0.95	1.5	25	185	30	25	185	
1,1-Dichloroethene	3	0.59	1.5	65	135	30	65	135	
Acetone	10	1.9	5	20	160	30	20	160	
Carbon disulfide	3	0.86	1.5	45	160	30	45	160	
Methylene chloride	3	0.75	1.5	55	140	30	55	140	
trans-1,2-Dichloroethene	3	0.56	1.5	65	135	30	65	135	
1,1-Dichloroethane	3	0.32	1.5	75	125	30	75	125	
2,2-Dichloropropane	3	0.57	1.5	65	135	30	65	135	
cis-1,2-Dichloroethene	3	0.39	1.5	65	125	30	65	125	
2-Butanone	10	1.8	5	30	160	30	30	160	
Bromochloromethane	3	0.36	1.5	70	125	30	70	125	
Chloroform	3	0.29	1.5	70	125	30	70	125	
1,1,1-Trichloroethane	3	0.55	1.5	70	135	30	70	135	
Carbon tetrachloride	3	0.87	1.5	65	135	30	65	135	
1,1-Dichloropropene	3	0.55	1.5	70	135	30	70	135	
Benzene	3	0.49	1.5	75	125	30	75	125	
1,2-Dichloroethane	3	0.28	1.5	70	135	30	70	135	
Trichloroethene	3	0.38	1.5	75	125	30	75	125	
1,2-Dichloropropane	3	0.41	1.5	70	120	30	70	120	
Dibromomethane	3	0.42	1.5	75	130	30	75	130	
Bromodichloromethane	3	0.32	1.5	70	130	30	70	130	
cis-1,3-Dichloropropene	3	0.79	1.5	70	125	30	70	125	
4-Methyl-2-pentanone	10	0.63	5	45	145	30	45	145	
Toluene	3	0.54	1.5	70	125	30	70	125	
trans-1,3-Dichloropropene	3	0.57	1.5	65	125	30	65	125	
1,1,2-Trichloroethane	3	0.29	1.5	60	125	30	60	125	
Tetrachloroethene	3	0.54	1.5	65	140	30	65	140	

Target Analyte	RL	MDL	Blank	MS/MSD			BS/BSD		
				LCL	UCL	RPD	LCL	UCL	RPD
2-Hexanone	10	0.59	5	45	145	30	45	145	
1,3-Dichloropropane	3	0.6	1.5	75	125	30	75	125	
1,2-Dibromoethane	3	1.1	1.5	70	125	30	70	125	
Chlorobenzene	3	0.4	1.5	75	125	30	75	125	
Ethylbenzene	3	0.73	1.5	75	125	30	75	125	
1,1,1,2-Tetrachloroethane	3	0.75	1.5	75	125	30	75	125	
m,p-Xylene	6	1.5	1.5	80	125	30	80	125	
o-Xylene	3	0.66	1.5	75	125	30	75	125	
Styrene	3	0.51	1.5	75	125	30	75	125	
Bromoform	3	0.74	1.5	55	135	30	55	135	
Isopropylbenzene	3	0.73	1.5	75	130	30	75	130	
1,1,2,2-Tetrachloroethane	3	0.67	1.5	55	130	30	55	130	
n-Propylbenzene	3	0.53	1.5	65	135	30	65	135	
Bromobenzene	3	0.76	1.5	65	120	30	65	120	
1,2,3-Trichloropropane	3	0.34	1.5	65	130	30	65	130	
2-Chlorotoluene	3	0.57	1.5	70	130	30	70	130	
1,3,5-Trimethylbenzene	3	0.66	1.5	65	135	30	65	135	
4-Chlorotoluene	3	0.71	1.5	75	125	30	75	125	
tert-Butylbenzene	3	0.58	1.5	65	130	30	65	130	
1,2,4-Trimethylbenzene	3	0.65	1.5	65	135	30	65	135	
sec-Butylbenzene	3	0.56	1.5	65	130	30	65	130	
4-Isopropyltoluene	3	0.54	1.5	75	135	30	75	135	
1,3-Dichlorobenzene	3	0.56	1.5	70	125	30	70	125	
1,4-Dichlorobenzene	3	0.7	1.5	70	125	30	70	125	
n-Butylbenzene	3	0.58	1.5	65	140	30	65	140	
1,2-Dichlorobenzene	3	0.55	1.5	75	120	30	75	120	
1,2-Dibromo-3-chloropropane	3	1.2	1.5	40	135	30	40	135	
1,2,4-Trichlorobenzene	3	0.48	1.5	65	130	30	65	130	
Hexachlorobutadiene	3	0.66	1.5	55	140	30	55	140	
Naphthalene	3	0.46	1.5	40	125	30	40	125	
1,2,3-Trichlorobenzene	3	0.66	1.5	60	135	30	60	135	

Medium level soil

Target Analyte	RL	MDL	Blank	MS/MSD			BS/BSD		
				LCL	UCL	RPD	LCL	UCL	RPD
Dibromochloromethane	100	11	50	65	130	30	65	130	
Dichlorodifluoromethane	100	43	50	35	135	30	35	135	
Chloromethane	100	26	50	50	130	30	50	130	
Vinyl chloride	100	34	50	60	125	30	60	125	
Bromomethane	100	26	50	30	160	30	30	160	
Chloroethane	100	21	50	40	155	30	40	155	
Trichlorofluoromethane	100	26	50	25	185	30	25	185	
1,1-Dichloroethene	100	16	50	65	135	30	65	135	
Acetone	500	50	250	20	160	30	20	160	
Carbon disulfide	100	5.6	50	45	160	30	45	160	
Methylene chloride	100	160	50	55	140	30	55	140	
trans-1,2-Dichloroethene	100	14	50	65	135	30	65	135	
1,1-Dichloroethane	100	13	50	75	125	30	75	125	
2,2-Dichloropropane	100	20	50	65	135	30	65	135	
cis-1,2-Dichloroethene	100	12	50	65	125	30	65	125	
2-Butanone	500	61	250	30	160	30	30	160	
Bromochloromethane	100	19	50	70	125	30	70	125	
Chloroform	100	13	50	70	125	30	70	125	
1,1,1-Trichloroethane	100	21	50	70	135	30	70	135	
Carbon tetrachloride	100	22	50	65	135	30	60	135	
1,1-Dichloropropene	100	16	50	70	135	30	70	135	
Benzene	100	6.6	50	75	125	30	75	125	
1,2-Dichloroethane	100	12	50	70	135	30	70	135	
Trichloroethene	100	18	50	75	125	30	75	125	
1,2-Dichloropropane	100	11	50	70	120	30	70	120	
Dibromomethane	100	14	50	75	130	30	75	130	
Bromodichloromethane	100	12	50	70	130	30	70	130	
cis-1,3-Dichloropropene	100	12	50	70	125	30	70	125	
4-Methyl-2-pentanone	500	14	250	45	145	30	45	145	
Toluene	100	8.7	50	70	125	30	70	125	
trans-1,3-Dichloropropene	100	9.2	50	65	125	30	65	125	
1,1,2-Trichloroethane	100	6.8	50	60	125	30	60	125	
Tetrachloroethene	100	17	50	65	140	30	65	140	
2-Hexanone	500	7	250	45	145	30	45	145	

Target Analyte	RL	MDL	Blank	MS/MSD			BS/BSD		
				LCL	UCL	RPD	LCL	UCL	RPD
1,3-Dichloropropane	100	15	50	75	125	30	75	125	
1,2-Dibromoethane	100	14	50	70	125	30	70	125	
Chlorobenzene	100	11	50	75	125	30	75	125	
Ethylbenzene	100	6.4	50	75	125	30	75	125	
1,1,1,2-Tetrachloroethane	100	8.6	50	75	125	30	75	125	
m,p-Xylene	200	11	100	80	125	30	80	125	
o-Xylene	100	15	50	75	125	30	75	125	
Styrene	100	7.4	50	75	125	30	75	125	
Bromoform	100	20	50	55	135	30	55	135	
Isopropylbenzene	100	9.4	50	75	130	30	75	130	
1,1,2,2-Tetrachloroethane	100	15	50	55	130	30	55	130	
n-Propylbenzene	100	9	50	65	135	30	65	135	
Bromobenzene	100	9.9	50	65	120	30	65	120	
1,2,3-Trichloropropane	100	29	50	65	130	30	35	130	
2-Chlorotoluene	100	7.9	50	70	130	30	70	130	
1,3,5-Trimethylbenzene	100	9	50	65	135	30	65	135	
4-Chlorotoluene	100	10	50	75	125	30	75	125	
tert-Butylbenzene	100	11	50	65	130	30	65	130	
1,2,4-Trimethylbenzene	100	6	50	65	135	30	65	135	
sec-Butylbenzene	100	10	50	65	130	30	65	130	
4-Isopropyltoluene	100	9.4	50	75	135	30	75	135	
1,3-Dichlorobenzene	100	15	50	70	125	30	70	125	
1,4-Dichlorobenzene	100	5.4	50	70	125	30	70	125	
n-Butylbenzene	100	8.3	50	65	140	30	65	140	
1,2-Dichlorobenzene	100	15	50	75	120	30	75	120	
1,2-Dibromo-3-chloropropane	100	23	50	40	135	30	40	135	
1,2,4-Trichlorobenzene	100	20	50	65	130	30	65	130	
Hexachlorobutadiene	100	21	50	55	140	30	55	140	
Naphthalene	100	18	50	40	125	30	40	125	
1,2,3-Trichlorobenzene	100	24	50	60	135	30	60	135	

Surrogate recovery limits for soils and waters

Surrogates Water	LCL	UCL
Dibromofluoromethane	85	115
1,2-Dichloroethane-d4	70	120
Toluene-d8	85	120
4-Bromofluorobenzene	75	120

Surrogate Soil	LCL	UCL
Dibromofluoromethane	80	123
1,2-Dichloroethane-d4	62	134
Toluene-d8	85	115
4-Bromofluorobenzene	85	120

*These control limits were taken from the DoD QSM and are subject to change.
Not all analytes have a second source for LCS and MS/MSD

APPENDIX XIV

Agency Requirements

	DOD	AFCEE	USACE		SW 846
QC Check	**Acceptance Criteria**	**Acceptance Criteria**	**Acceptance Criteria**	**Sporadic Marginal Failure**	**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.	RL>3xMDL; MDL check sample (~2xMDL) quarterly		
BFB Tuning	Refer to method	mass 50 : 15%-40% of mass 95	mass 50 : 15%-40% of mass 95		mass 50 : 15%-40% of mass 95
		mass 75 : 30%-60% of mass 95	mass 75 : 30%-60% of mass 95		mass 75 : 30%-60% of mass 95
		mass 95 : base peak, 100%	mass 95 : base peak, 100%		mass 95 : base peak, 100%
		mass 96 : 5%-9% of mass 95	mass 96 : 5%-9% of mass 95		mass 96 : 5%-9% of mass 95
		mass 173 : < 2% of mass 174	mass 173 : < 2% of mass 174		mass 173 : < 2% of mass 174
		mass 174 : > 50% of mass 95	mass 174 : > 50% of mass 95		mass 174 : > 50% of mass 95
		mass 175 : 5%-9% of mass 174	mass 175 : 5%-9% of mass 174		mass 175 : 5%-9% of mass 174
		mass 176 : 95%-101% of mass 174	mass 176 : 95%-101% of mass 174		mass 176 : 95%-101% of mass 174
		mass 177 : 5%-9% of mass 176	mass 177 : 5%-9% of mass 176		mass 177 : 5%-9% of mass 176
					OR criteria specified by MS instrument manufacturer
Minimum Five Point Initial Calibration	1. SPCC RF for chlorobenzene and 1,1,2,2-tetrachlorolethane > 0.30, for chloromethane, bromoform, and 1,1-dichloroethane > 0.1	1. SPCC: Average RF > 0.30, for chloromethane, bromoform, and 1,1-dichloroethane > 0.1	1. SPCC minimum RF per method requirements		1. SPCC RF for chlorobenzene and 1,1,2,2-tetrachlorolethane > 0.30, for chloromethane, bromoform, and 1,1-dichloroethane > 0.1
	2. CCC %RSD < 30%	2. CCC %RSD < 30%	2. CCC %RSD < 30%		2. CCC %RSD < 30%
	3. And one of the options below	3. And one of the options below	Primary Evaluation:		3. And one of the options below

	DOD	AFCEE	USACE		SW 846
QC Check	**Acceptance Criteria**	**Acceptance Criteria**	**Acceptance Criteria**	**Sporadic Marginal Failure**	**Acceptance Criteria**
	Option 1: Each Analyte RSD < 15%	Option 1: Averaged - Grand mean RSD < 15% and no RSD > 30%	%RSD < 15%, r > 0.995; r2 > 0.990		Option 1: Averaged - RSD < 15%
	Option 2: Averaged - Grand mean RSD < 15% and no RSD > 30%	Option 2: Linear - r > 0.995	Alternative Evaluation:	Alternative Evaluation:	Option 2: Linear - r > 0.99
	Option 3: Linear - r > 0.995	Option 3: Non-linear - r2 > 0.990, minimum of 6 pts for 2nd order, 7pts for 3rd order	Mean %RSD for all target analytes <15%, with maximum allowable restriction noted at right for individual analytes.	Maximum allowable %RSD for each individual target analyte < 30%.	Option 3: Non-linear - r2 > 0.99, minimum of 6 pts for 2nd order, 7pts for 3rd order
	Option 4: Non-linear - r2 > 0.990, minimum of 6 pts for 2nd order, 7pts for 3rd order				AND %D < 20% at each calibration level recommended
Second Source	%D < 25%	%D < 25%	%D < 20%		
Relative Retention Times	RRT of each analyte within + 0.06 RRT units	RRT of each analyte within + 0.06 RRT units			RRT of each analyte within + 0.06 RRT units
Continuing Calibration Verification	1. SPCC RF for chlorobenzene and 1,1,2,2-tetrachlorolethane > 0.30, for chloromethane, bromoform, and 1,1-dichloroethane > 0.1	1. SPCC RF for chlorobenzene and 1,1,2,2-tetrachlorolethane > 0.30, for chloromethane, bromoform, and 1,1-dichloroethane > 0.1	1. SPCC : minimum RF per method requirements		1. SPCC RF for chlorobenzene and 1,1,2,2-tetrachlorolethane > 0.30, for chloromethane, bromoform, and 1,1-dichloroethane > 0.1
	2. CCC %D < 20%	2. CCC %D < 20%	2. CCC %D < 20%		2. CCC < 20%
	3. Grand mean %D < 20% and no %D > 25%	3. %D < 20%	3. %D < 20% for project-specified contaminants of concern.		
	Every 12 hours	Every 12 hours	Every 12 hours		Every 12 hour analytical shift

	DOD	AFCEE	USACE		SW 846
QC Check	**Acceptance Criteria**	**Acceptance Criteria**	**Acceptance Criteria**	**Sporadic Marginal Failure**	**Acceptance Criteria**
Calibration Verification Internal Standard	Retention time within + 30 sec from ICAL. EICP are within -50% to 100% of ICAL.	Retention time within + 30 sec from ICAL. EICP are within -50% to 100% of ICAL.			Retention time within + 30 sec from ICAL. EICP are within -50% to 100% of ICAL.
Method Blank	No analytes > 1/2 RL. No common laboratory contaminant analytes > RL	No analytes > RL	No analytes > 1/2 RL. No common laboratory contaminant 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	Water: 80%-120% recovery	60%-140% recovery	Lab-derived
			Soil: 75%-125% recovery		
Matrix Spike	Same as LCS	Same as LCS	70%-130% recovery	60%-140% recovery	Lab-derived
Matrix Spike Duplicate	RPD < 30%	Water: RPD < 20%	Water: RPD < 30%	Water: RPD < 40%	Lab-derived
		Soil: RPD < 30%	Soil: No RPD limits	Soil: No RPD limits	
Surrogate Spike	Water:	Water:	Interference-free matrix:		Lab-derived
	1,2-dichloroethane-d4: 70-120%	1,2-dichloroethane-d4: 72-119%	Water: 80-120%		
	4-bromo-fluorobenzene: 75-120%	4-bromo-fluorobenzene: 76-119%	Soil: 75-125%		
	Dibromo-fluoromethane: 85-115%	Dibromo-fluoromethane: 85-115%			
	toluene-d8: 85-120%	toluene-d8: 81-120%	Project sample matrix:		
	Soil:	Soil:	70-130%		

	DOD	AFCEE	USACE		SW 846
QC Check	**Acceptance Criteria**	**Acceptance Criteria**	**Acceptance Criteria**	**Sporadic Marginal Failure**	**Acceptance Criteria**
	1,2-dichloro-ethane-d4: lab-derived	1,2-dichloro-ethane-d4: 52-149%			
	4-bromo-fluorobenzene: 85-120%	4-bromo-fluorobenzene: 84-118%			
	Dibromo-fluoromethane: lab-derived	Dibromo-fluoromethane: 65-135%			
	toluene-d8: 85-115%	toluene-d8: 84-116%			
Results reported between MDL and RL	Apply J flags	Apply F flags	Apply J flags		
Allowable Sporadic Marginal Excedence/Failure	> 90 analytes - 5		91 - 105 analytes - 7		
	71 - 90 analytes - 4		76 - 90 analytes - 6		
	51 - 70 analytes - 3		61 - 75 analytes - 5		
	31 - 50 analytes - 2		46 - 60 analytes - 4		
	11 - 30 analytes - 1		31 - 45 analytes- 3		
	< 11 analytes - 0		16 - 30 analytes - 2		
			5 - 15 analytes - 1		